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**ELEMENTARY PRACTICAL  
METALLURGY**



# ELEMENTARY PRACTICAL METALLURGY FOR TECHNICAL STUDENTS & OTHERS

BY

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With 25 Illustrations



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## PREFACE

As the title indicates, this book has been prepared for the use of those who wish to obtain a practical knowledge of the common metals by means of a laboratory course. Many years' experience of the work carried on in a large laboratory has made it evident that the majority of students attending technical courses, and in some cases college courses, desire only so much practical instruction as will give them an insight into the properties of the metals in which they are interested. Even at the beginning of their work, many desire to specialize in certain directions. Thus, out of 92 students who presented themselves in one session, 10 were interested in ironfounding, 22 in iron and steel, 11 in gold and silver, 23 in brass and similar alloys, and 26 in general metallurgy. It is quite evident from this that any single course would not adequately meet such diverse wants ; and several courses have been organized to meet the demand.

A general review of the book will make it clear that laboratory courses can be arranged for each section of workers. In fact it has been written up from the practical notes of special courses which have been developing for several years to meet the requirements of the various metal trades.

There are chapters on parts of the subject which are

of general interest, such as fuel, refractory materials, and mechanical testing. Others deal with certain chemical changes which should be studied; while others again deal with iron and steel, metals and alloys, and gold and silver. Thus, for example, a student engaged in the engineering trades may go through a series of experiments in iron and steel and such non-ferrous alloys as he may meet with in practice. He may also obtain a knowledge of fuel and refractory materials. The same applies to other trade courses. The book also offers sufficient scope for a course in general metallurgy extending over two years; so that those who wish to obtain a wider view of the subject will find in it all that is necessary until they reach the higher stages.

The text is largely devoted to instructions for carrying out the experiments, but a sufficient amount of descriptive matter is introduced to render the practical work intelligible. At the same time it is not intended to supplant the theoretical teaching which accompanies the practical work.

The author is indebted to Mr. E. H. Stansbie for reading the proofs, and for assistance with the drawings. His thanks are also due to Mr. W. H. Juggins for his help with the drawings.

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## CHAPTER I

### INTRODUCTORY

THE study of the common metals and their alloys by carefully arranged experiments in the laboratory is of great use in preparing the metal worker for the intelligent treatment of the various metals and alloys used in the arts and manufactures. Information thus acquired is often of much value in overcoming difficulties met with in working such metals as are abnormal in character due either to impurities present or to improper treatment. Further, the experience gained in carrying out the experiments successfully is of much value from other than the purely commercial point of view.

The experiments described in the following pages require only the apparatus and materials found in a metallurgical laboratory with the ordinary equipment.

**The Wind Furnace**, which is easily managed, is shown in Fig. 1. It consists of an iron case lined with fire bricks and fitted with loose fire-bars. The top is covered during working with fire-bricks strengthened by iron clamps running lengthwise and curved in the middle to facilitate the removal of the cover. The fire-space is 12 in. square by 18 in. deep. The horizontal flue connected with the main flue is controlled by a damper. With a good draught, good coke, and careful stoking a temperature of  $1450^{\circ}$  C. (the melting-point of nickel)

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can be obtained. A furnace of the dimensions given will hold three or four of the small crucibles generally used for experimental work. Smaller furnaces are

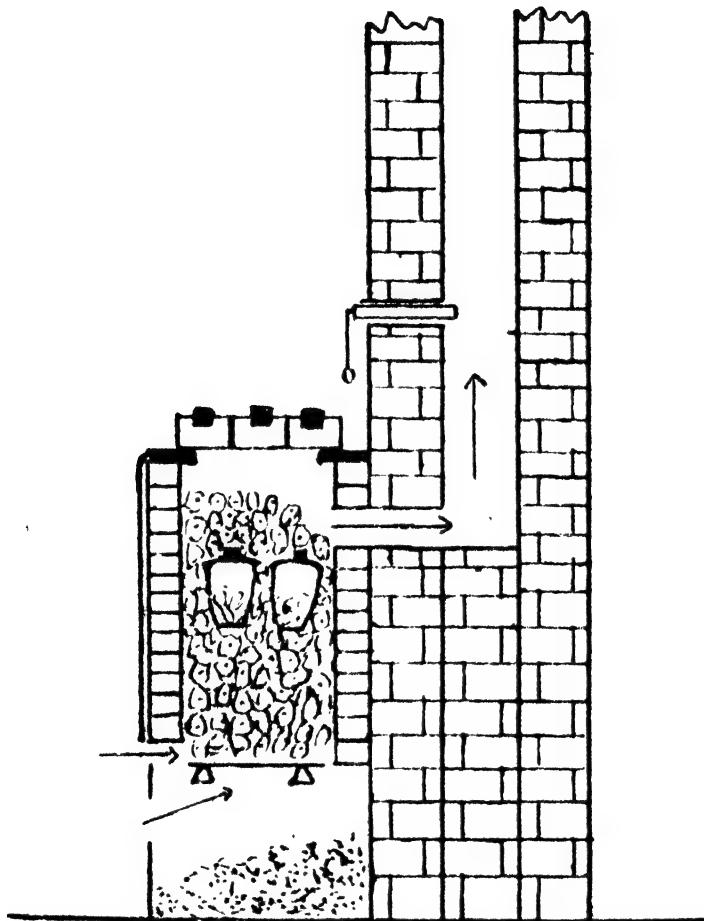


FIG. 1

sometimes built, but they will only take one pot conveniently.

When only a comparatively short time is set apart for laboratory work the fire should be ready, and the student is advised to see that it is in good condition before preparing his experiments, and to keep it properly

coked in the intervals. By doing so, much time is saved, and the work goes on smoothly. The best position for the crucible is towards the back of the fire and just below the flue. For melting the more refractory metals, the pot should be covered with a layer of coke of medium size, but the furnace should not be filled up to the cover.

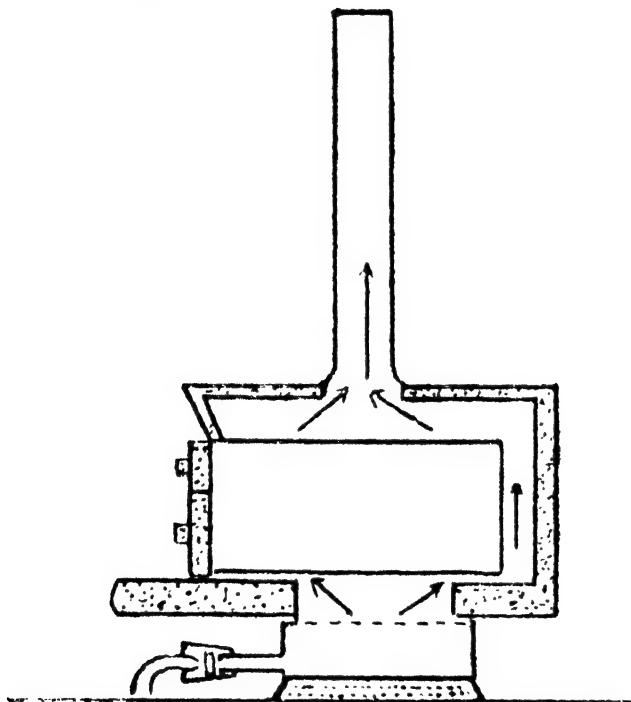


FIG. 2

**The Muffle Furnace** (Fig. 2). This is simply a fire-clay chamber heated from outside, and as it is used mostly for experiments in which a good supply of air is needed, the back of the chamber is usually pierced by two or three holes in the upper part, which very much facilitate the passage of a current of air through it. The coke muffle, which is still used for special

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work, has been largely superseded by the gas muffle for the heating of which coal gas is used.

**The Injector Furnace.** A temperature sufficient for many experiments can be maintained in this furnace, which is based upon the principle of the blowpipe. The crucible stands in a circular fire-clay chamber, and the nose of the injector is inserted in a hole in the side. The air-blast is supplied by a foot blower. It requires personal attention during the whole time, and on that account is not so convenient as the wind furnace. It

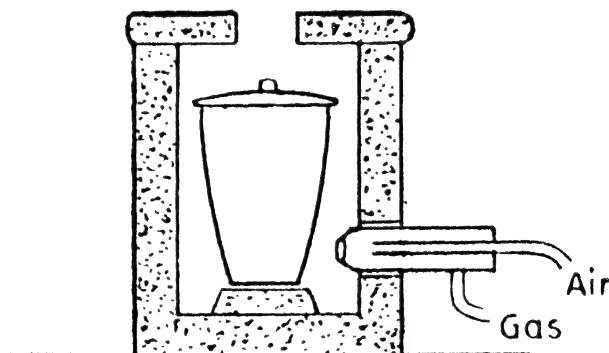


FIG. 3

is useful, however, when a single experiment has to be carried through, and for special work. The general arrangement is made clear by Fig. 3.

**The Meker Furnace.** The burner for this furnace is on the Bunsen principle, but is fitted at the top with a cap containing a nickel grating about  $\frac{1}{2}$  in. deep and with 100 channels to the square inch. The length and cross-section of these channels are so adjusted that a good body of Bunsen flame is obtained in which the combustion is more uniformly distributed than in the ordinary flame. The furnace is shown in section in Fig. 4. A temperature of about  $950^{\circ}\text{C}$ . can be obtained

inside a clay crucible. The burner can be fitted with an inlet by which oxygen under pressure may be used, and a higher temperature obtained.

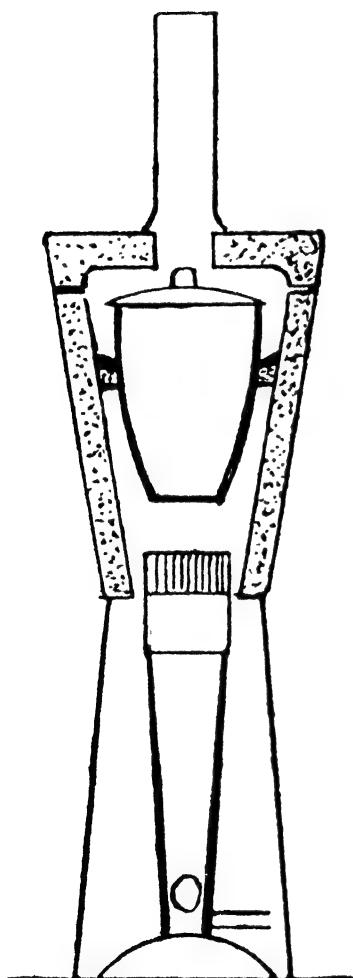


FIG. 4

**The Bunsen Burner.** One form of burner used for bench experiments is shown in Fig. 5, and another in Fig. 15. In principle it is an inversion of the injector in that the rush of gas from a fine jet draws in sufficient air to form a mixture for complete combustion. The quantity of air drawn in is regulated by the size of air-ports opening near the jet.

**The Hot Plate.** This is an iron plate standing on legs with one or more Bunsen burners

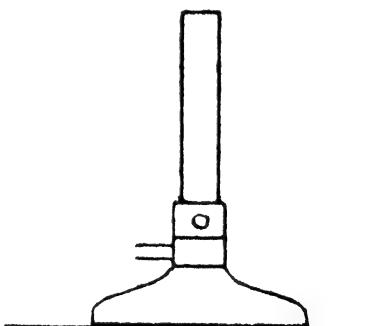


FIG. 5

underneath. The plate may be covered with sand if required. It is useful for heating beakers, drying precipitates, and other materials. It should stand in a well-ventilated fume-closet.

**Crucibles** (Fig. 6). Clay crucibles are the most

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generally useful, and are made in various sizes for experimental purposes. Graphite crucibles are very suitable for melting steel and cast-iron, and for making brasses. Morgan's salamander pots are durable, and give good results. They are glazed to prevent the absorption of moisture. Crucibles made from the same material as arc-carbons are not wetted by molten sulphides, and are excellent for making small quantities of gold and silver alloys, but they are now difficult to

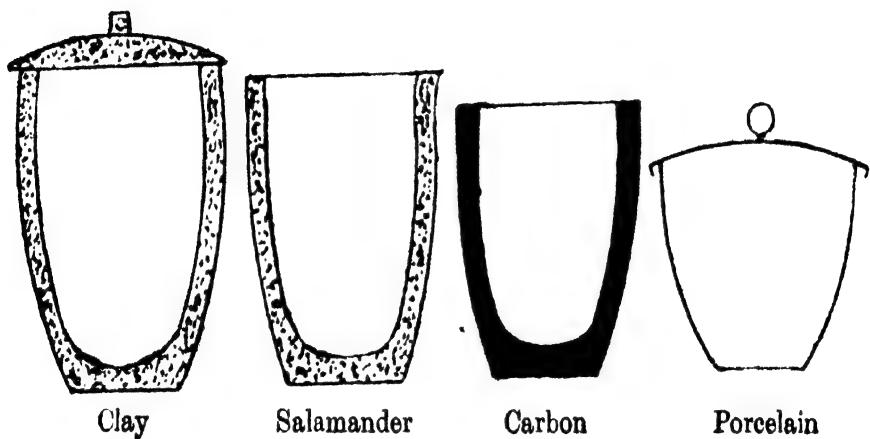


FIG. 6.—Crucibles

obtain as the demand for them does not warrant their regular manufacture. The *brasqued* crucible is simply a clay one lined with carbon. Charcoal powder made cohesive but not sticky with a mixture of treacle and water is well pressed on the inside of the crucible, then trimmed and dried. The crucible, closed with a well-fitting lid, is then heated in a muffle to make the lining firm.

**Roasting Dishes** (Fig. 7). These are thin, shallow clay dishes, and are used for muffle experiments in which the materials do not fuse.

**Scorifiers** are thick clay dishes for experiments in which molten oxides are formed. The clay furnishes silica to form a scoria with the oxides.

**Cupels.** These small vessels were formerly made from bone ash moistened with a little water, but now

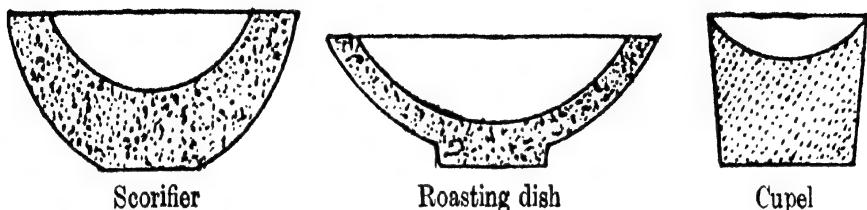


FIG. 7

Portland cement is the important constituent of commercial cupels.

**Tongs.** There is quite an assortment of appliances for working with the furnace and muffle, but three sizes of tongs are sufficient for ordinary work. Furnace tongs should have handles about 26 in. long for

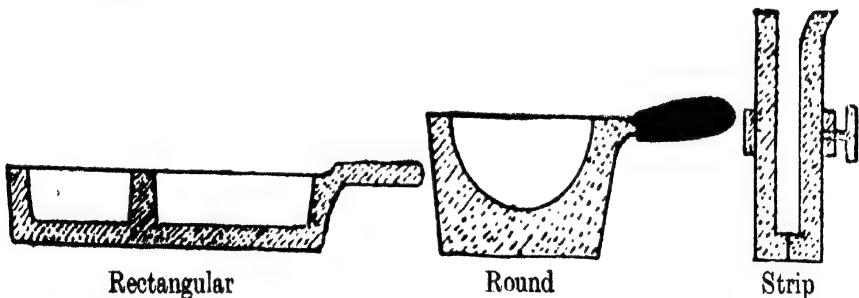


FIG. 8.—Moulds

convenient working; while tongs with handles about 16 in. are more suitable for muffle work; and for carrying hot vessels, ingots, etc., handles of about 9 in. are convenient.

**Iron Moulds.** Open moulds are of two kinds, rectangular and round. Convenient dimensions for the

most commonly used rectangular moulds are  $2\frac{1}{2}$  in. by  $\frac{5}{8}$  in. by  $\frac{1}{2}$  in. deep and  $1\frac{1}{2}$  in. by  $\frac{3}{4}$  in. by  $\frac{5}{8}$  in. deep. The round mould is about  $1\frac{3}{4}$  in. in diameter, and is sometimes described as the button mould from the shape of the cast. Strip and rod moulds are in two parts, which can be clamped together for use. The general form of these moulds is seen in Fig. 8.

**The Balance.** The metric system of weights is used

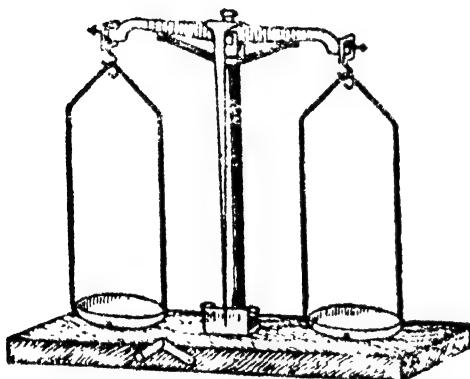


FIG. 9

in the weighing operations, but it is so simple that the student who has not yet had to think in grams will find no difficulty in its use. A suitable set contains weights from 100 grams to  $\frac{1}{1000}$  gram, or 1 milligram. The fractions of the gram are the decigram  $\frac{1}{10}$ , the centigram

$\frac{1}{100}$ , and the milligram  $\frac{1}{1000}$  of a gram. The gram equals 15.432 grains. The balance used for fine weighing should turn readily with 1 milligram. The form shown in Fig. 9 is sufficiently sensitive for the work to be described later. A stronger balance turning with a decigram is required for weighing materials for crucible charges. Weighings should not be made on the bare scale-pan. A watch-glass counterpoised with a piece of lead should be kept for the sensitive balance, and two pieces of glazed paper for the other. This is especially convenient in weighing powders.

**Measuring Vessels.** As it is necessary to take measured volumes of various liquids for some experiments, a short

description of the principal measures will be useful. The metric system is again called upon to supply the standard volume. This is the cubic centimetre, which is defined as the volume occupied by 1 gram of water at its point of maximum density, *i.e.*  $4^{\circ}\text{C}$ . The cubic decimetre or litre equals 1000 cubic centimetres. It is usual to speak in terms of cubic centimetres, the contraction for which is c.c.

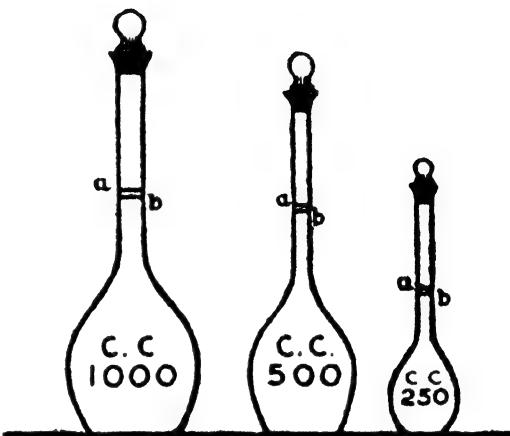


FIG. 10

Three measuring flasks are shown in Fig. 10. If

a liquid is poured into the dry flask up to the mark *b* its volume is equal to that marked on the flask providing the temperature is the same as that at which the flask was graduated. This is the average laboratory temperature,  $15^{\circ}\text{ C.}$ , and slight variations from it do not affect ordinary measurements. If

more liquid is added so as to bring it up to the mark *a* the flask will deliver the volume marked on it.

Three measuring cylinders and a pipette are shown in Fig. 11. With these, volumes from 500 c.c. to  $\cdot 1$  c.c. are readily measured. These vessels are graduated to

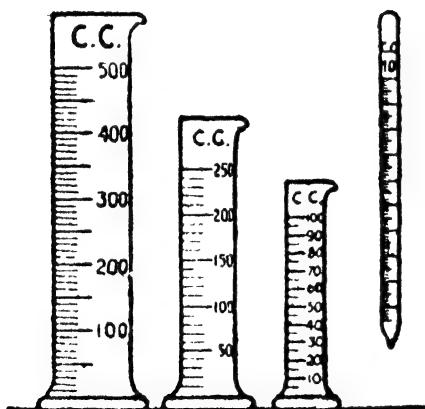


FIG. 11

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deliver. To use the pipette the point is immersed, and the liquid sucked into the pipette by the mouth. The finger is then placed on the open end, and the excess liquid allowed to run out. Care must be taken not to suck the liquid into the mouth. The quantity delivered is regulated by easing the finger.

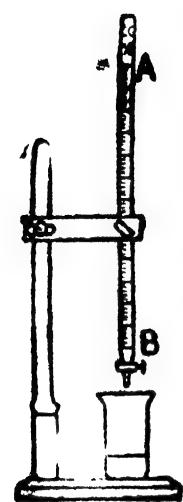


FIG. 12

**The Burette.** This measure is capable of giving accurate results with careful manipulation. It is shown in Fig. 12, and consists of a long graduated tube A fitted with a tap B. The common form has a capacity of 50 c.c. and is graduated in tenths of a c.c. It is supported in a vertical position, as shown. The surface of the liquid in the burette is curved, and the readings are best made by reference to the lower or convex surface.

To use the burette, it is washed with distilled water, rinsed with a little of the liquid to be measured, and filled above the zero mark. The tap is then opened for the nozzle to fill, and the liquid is allowed to escape drop by drop until the convex surface exactly coincides with the zero mark. After this the volume of liquid allowed to escape can be measured to .05 c.c.

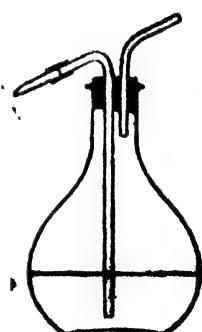


FIG. 13

**The Wash-bottle.** A common form of wash-bottle is shown in Fig. 13. It is fitted with a flexible nozzle, so that by blowing down the short tube a stream of water can be directed in any direction. The flask is thin, and the water in it can be heated

on the hot plate, or on gauze over the Bunsen burner.

**The Filter Funnel.** This serves as a support for a filter-paper when used for collecting a precipitate, or for separating suspended solid matter from a solution. The funnel and stand are shown in Fig. 14. The filter-paper is folded on itself twice, and then opened out to form a cone with three thicknesses on one side and one on the other. It is arranged in the funnel and wetted to cause it to cling to the side. In filtering, the liquid is poured from the beaker or other vessel down a glass rod into the filter. Any solid matter left in the beaker is washed on to the filter by means of a jet of water from the wash-bottle. The solid on the filter is well washed with water to displace the solution from which it has been separated.

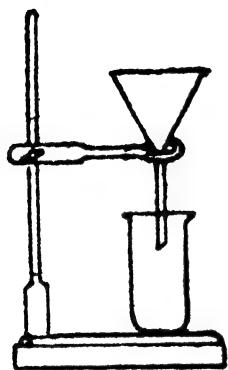


FIG. 14

**The Drying Cone.** This is simply a sheet-metal cone open at both top and bottom. It is very useful for drying precipitates, and may be placed on the hot plate, or over a Bunsen burner, as shown in Fig. 15.

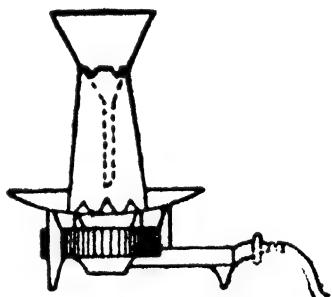


FIG. 15

**The Drying Oven.** This is a square copper box fitted with a swing door, and having a circular neck in the top, which is closed by a cork through which a thermometer passes to register the temperature of the interior. It

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is supported on a stand to allow of a Bunsen burner being placed under the bottom to heat it. Materials to be heated are placed on a shelf well above the bottom.

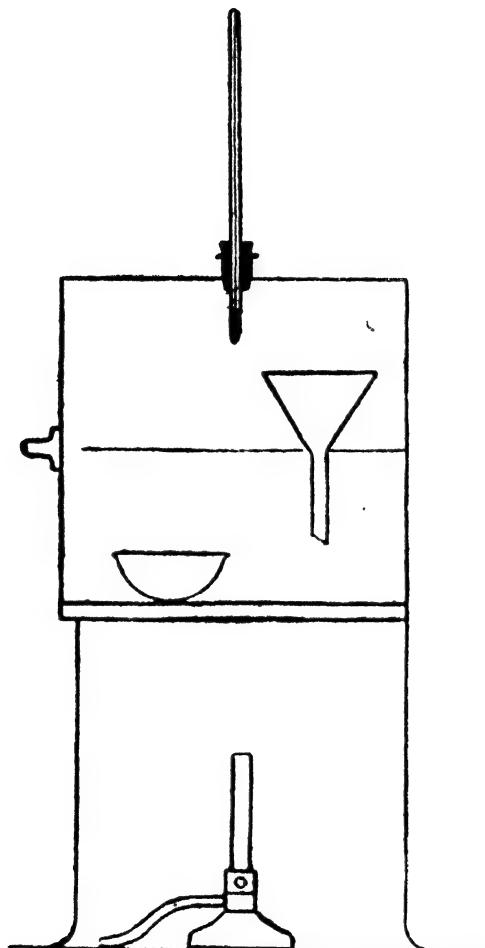


FIG. 16

A definite temperature is readily maintained by regulating the flame from the burner. The general form is shown in Fig. 16.

The following small apparatus is usually supplied to each student :

Small hammer.	Glass funnel.
," steel anvil.	," beakers.
Spatula.	," rod.
Shears.	," test-tubes and rack.
Pliers.	Evaporating basin.
Brass forceps.	Porcelain crucible.
Small crucible tongs.	Mortar and pestle.
Camel-hair brush.	Glazed paper.
Wire triangle.	Filter papers.

The student should provide himself with a pocket lens, or a watchmaker's lens, for examining fractures.

The various pieces of apparatus about the laboratory and not mentioned above will become familiar to the student by use.

**General Principles.** The great variety of substances, which make up the solid crust of the earth together with the water and air upon it, are composed of a few simple substances called elements. Some of these elements are quite common, others are very rare ; and others again take an intermediate place.

The name element suggests something simple in character and definite in properties, and this is so. Thus an element may be described as a substance which has resisted all attempts to obtain more than one kind of material from it, and which possesses properties enabling it to be recognized and distinguished from all other substances. Thus copper is copper and nothing more.

The elements are divided more or less sharply into metals and non-metals, but there is no difficulty in distinguishing those given in the Table on page 15. It will be necessary to use certain proportional numbers or

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weights which are characteristic of the various elements, and which are called their *equivalent* or *combining weights*. These are intimately associated with another set of numbers which are either the same as, or simple multiples of, the first set. These are called *atomic weights*. It is really of no importance which numbers are used in practical work as long as a distinction is drawn between them. The atomic weights of the various elements are represented by symbols such as O for 16 parts by weight of oxygen, Cu for 63.6 parts by weight of copper, and H for 1 part by weight of hydrogen; the latter being the lightest substance known. It is made up of the lightest atoms, and is taken as the standard of reference. The atom is the smallest indivisible particle of an element, and since it is matter, however small a particle it may be, it possesses that universal property of matter, which confers upon it its weight. From the practical point of view, however, these atomic weights represent actual quantities, which can be handled in the formation and decomposition of compounds containing the elements they refer to.

It will be noticed that there is a simple relation between the equivalent and atomic weights given in the Table. If the equivalent weight of the element is multiplied by one of the whole numbers, 1, 2, 3, 4, its atomic weight will result. The whole number which is to be used is readily seen. The equivalent weights are obtained by experiments and represent the weights of the respective elements which will combine with 1 part by weight of hydrogen, or 35.5 parts by weight of chlorine. Hydrogen is the standard for the non-metals, and chlorine for the metals. In a few cases

there are two or more equivalents for the same element ; this being due to the fact that two or more compounds are formed with the standard element. In these cases

COMMON METALS							
Name	Symbol	Equivalent weight	Atomic weight	Name	Symbol	Equivalent weight	Atomic weight
Aluminium	Al	9	27.1	Manganese	Mn	27.4	54.9
Antimony	Sb	40	120.2	Mercury	Hg	100.3	200.6
Barium	Ba	68.7	137.4	Molybdenum	Mo	24	96.0
Bismuth	Bi	104	208.0	Nickel	Ni	29.3	58.7
Cadmium	Cd	56.2	112.4	Potassium	K	39.1	39.1
Calcium	Ca	20	40.0	Platinum	Pt	48.8	195.2
Chromium	Cr	17.3	52.0	Rhodium	Rh	34.3	103.0
Cobalt	Co	29.5	59.0	Silver	Ag	107.9	107.9
Copper	Cu	31.8	63.6	Sodium	Na	23	23.0
Gold	Au	65.7	197.2	Strontium	Sr	43.8	87.6
Iron	Fe	27.9	55.8	Tin	Sn	29.7	119.0
Iridium	Ir	48.3	193.1	Tungsten	W	46.2	184.8
Lead	Pb	103.5	207.1	Vanadium	V	17.1	51.4
Magnesium	Mg	12.1	24.3	Zinc	Zn	32.7	65.4

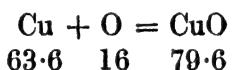
NON-METALS							
Arsenic	As	25	75	Iodine	I	126.9	126.9
Boron	B	3.7	11	Nitrogen	N	4.7	14
Bromine	Br	79.9	79.9	Oxygen	O	8	16
Carbon	C	3	12	Phosphorus	P	10.3	31
Chlorine	Cl	35.5	35.5	Sulphur	S	16	32
Fluorine	F	19	19	Selenion	Se	39.5	79
Hydrogen	H	1	1	Silicon	Si	7.1	28.3

the common equivalent only has been inserted in the Table.

The compounds form a second class of substances with definite and characteristic properties, and their number is legion. But if any definite one is singled out it is always found to contain the same elements in the same proportions, and to possess properties which

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distinguish it from all other compounds. It is quite immaterial how or whence it is obtained. Chemical combination between elements produces compounds, and these are just as definite as the elements themselves, but they can be decomposed and their elements obtained in the free state. In this they differ from elements. It is on account of the fixed law of chemical combination that it is possible to use such statements as :



and be quite certain that they will be borne out in practice whether the weights used are grams, ounces, pounds, or tons. Thus 63.6 tons of copper would combine with 16 tons of oxygen to form 79.6 tons of black copper oxide. These statements of chemical change are called chemical equations, and are freely used.

When the composition of a compound has been determined an arrangement of the symbols of its elements may be made to represent it. Thus  $\text{Al}_2\text{O}_3$  means

$\text{Al}_2\text{O}_3$ , or that 54 parts by weight of aluminium  $27 \times 2 + 16 \times 3$ , are combined with 48 parts by weight of oxygen to form 102 parts by weight of alumina. Two or more of these formula-weights may be required in indicating a chemical change, or in writing the formula of a more complex compound. This is shown by a number to the left of the formula, thus :  $2\text{Al}_2\text{O}_3$ , which means twice the full weight.

It will be at once recognized that every well-defined substance is not necessarily an element or a compound. There are very intimate mixtures of elements and compounds, and there are solutions which may be

either liquid or solid. But the proportions between the elements and compounds in a mixture may vary indefinitely; and those between the solvent and the dissolved substance may vary between wide limits. Thus two fine powders may be mixed in any proportion; and salt may be dissolved in water in any quantity up to the point of saturation.

It may be said then that the substances one has to deal with in practical work are elements, compounds, mixtures, and solutions.

**Common Fluxes and Reagents.** By far the greater number of inorganic compounds can be arranged under three heads: acids, bases, and salts, and as examples of all three are in constant use in the laboratory the important ones will be briefly described here.

**Acids.** All acids contain hydrogen which can be replaced by a metal, and a few contain only one other element in addition. But the majority contain oxygen as well as the other element, and are therefore more complex in composition.

**Hydrochloric Acid, HCl.** This useful liquid, which is known also as smoking salts and muriatic acid, is a solution of hydrochloric acid gas in water. The strongest acid contains about 40 per cent. HCl gas dissolved in 60 per cent. water. It can be diluted to any required strength. It dissolves some of the metals and most of the metallic oxides with formation of salts, namely **Chlorides**.

**Nitric Acid, HNO<sub>3</sub>.** This acid in the pure state is a strongly fuming, colourless liquid, which is more stable when mixed with water. The strongest commercial acid contains about 70 per cent. HNO<sub>3</sub> and 30 per cent. water. It mixes with water in all proportions. It

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dissolves most metals and oxides forming **Nitrates**. The common acid is called aqua-fortis.

**Sulphuric Acid**,  $H_2SO_4$ . The pure acid is a colourless, oily liquid which dissolves readily in water with evolution of much heat. In making the solution the acid should be poured slowly into the water contained in a vessel not readily cracked by heat. The commercial acid contains about 87 per cent.  $H_2SO_4$  and 13 per cent. water. The dilute solution dissolves some metals and many oxides with formation of **sulphates**. The acid is also known as oil of vitriol.

**Bases**. These are the oxides and hydroxides of the metals. If there are two oxides of the same metal, then the one containing the smaller proportion of oxygen is the more basic in character.

**Lime**, or Calcium Oxide,  $CaO$ , is a grey to white solid obtained by calcining limestone,  $CaCO_3$ , by which carbon dioxide is driven off and quicklime left. Lime absorbs moisture from the atmosphere and breaks down into powder, the oxide being converted into the hydroxide,  $CaH_2O_2$ . The water can be driven off again at a red heat. Although difficult to fuse it makes an excellent flux for some purposes.

**Magnesia**, or Magnesium Oxide,  $MgO$ , is obtained by calcining the carbonate  $MgCO_3$ . It is very refractory, does not absorb water, and is fluxing, but not as good as lime.

**Alumina**,  $Al_2O_3$ , Aluminium Oxide is a white powder, basic and very refractory. It is a common constituent of blast furnace flags, but is little used as a flux on the small scale.

**Litharge**, Lead Oxide,  $PbO$ . A yellow powder which fuses at a red heat. As the liquid solidifies it crystallizes.

It unites readily with silica at a red heat, forming a glass or silicate. Its fluxing power is very marked, but not often taken advantage of.

**Red Lead**,  $Pb_3O_4$ . When finely powdered litharge, or massicot, is heated to about  $300^{\circ} C.$  it absorbs oxygen and is converted into red lead. The extra oxygen is, however, given up at a higher temperature. It is used as a source of lead for dissolving gold and silver in ore assays, and in the treatment of residues containing gold and silver.

**Caustic Soda**, Sodium Hydroxide,  $NaOH$ . This compound is a white solid, readily soluble in water with evolution of heat. It reacts with acids, and exchanges its metal for the hydrogen of the acids with formation of salts, being itself converted into water. Thus with hydrochloric acid it forms common salt. It is a very powerful basic flux, but is not often used for that purpose. Its solution is used for removing grease from the surface of metals.

**Salts.** These are compounds containing the essential portions of acids and bases. The metal of the base takes the place of the hydrogen of the acid. In the case of the oxy-acids it is sometimes convenient to regard their salts as a combination of oxides. Thus limestone may be written as a compound of lime and carbon dioxide, thus  $CaO.CO_2$ , or it may be written  $CaCO_3$ . The former plan is usually adopted in the case of silicates; thus  $2CaO.SiO_2$  is a lime silicate.

**Common Salt**, Sodium Chloride,  $NaCl$ . This substance is sometimes used as a cover in melting operations to protect the contents of the crucible from the action of the atmosphere. It also has powerful fluxing properties with silica, converting it into a silicate. It

## 20 ELEMENTARY PRACTICAL METALLURGY

melts and volatilizes at a good red heat, and tends to remove those metals which form volatile chlorides, from a mixture of metals in the molten state.

**Fluorspar**, Calcium Fluoride,  $\text{CaF}_2$ . This compound is used to increase the fluidity of slags in pot assays. It seems to undergo very little change during the reactions.

**Sodium Carbonate**,  $\text{Na}_2\text{CO}_3$ , is an excellent basic flux, as the effective part of it is sodium oxide,  $\text{Na}_2\text{O}$ . The carbon dioxide may also exert an oxidizing action upon base metals, and thus assist their passage into the slag. The reduction of sulphides is also assisted by the desulphurizing action of the carbonate. It is used as a dry powder, which may be obtained by heating the crystallized salt,  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ , to remove the water of crystallization.

**Borax**,  $\text{Na}_2\text{B}_2\text{O}_7$ , is a white salt containing water of crystallization which causes the powder to swell when heated. For some purposes it is an advantage to remove this water by heating, thus obtaining calcined borax. It contains an excess of the acid oxide  $\text{B}_2\text{O}_3$  and so is able to dissolve metallic oxides (bases) and remove them. This is its function in hard soldering. It is a useful flux for various purposes.

**Tartar or Argol**,  $\text{KHC}_4\text{H}_4\text{O}_6$ , is impure potassium hydrogen tartrate. It is a product of the fermentation of grape juice, and varies from white to puce in colour. When heated it gives off a combustible gas, and leaves a residue of potassium carbonate,  $\text{K}_2\text{CO}_3$ , and carbon. It thus acts both as a fluxing and as a reducing agent.

**Nitre**, Saltpetre, or Potassium Nitrate,  $\text{KNO}_3$ . This compound is a very powerful oxidizing agent and is

commonly used as such; but it also furnishes an alkaline flux,  $K_2O$ .

**Calcium Sulphate**, Gypsum,  $CaSO_4$ , has been introduced into some of the fluxes for metal melting, but is not of much importance.

**Glass.** This is a solid solution of various silicates, and is capable of dissolving either metallic oxides or silica. It is sometimes used as a constituent in a fluxing mixture.

**Potassium Cyanide**,  $KCN$ , is a very powerful reducing agent, as it takes up oxygen from metallic oxides with formation of the cyanate,  $KCNO$ . It is a dangerous poison and should be handled carefully.

**Potassium Ferrocyanide**, or Yellow Prussiate of Potash,  $K_4FeC_6N_6$ , contains about 19 per cent. of carbon, much of which may be absorbed by iron at a red heat, or may be utilized as a reducing agent. It melts readily and is not poisonous when pure.

**Bisulphate of Potash**, Salenixum,  $KHSO_4$ , or the corresponding sodium salt, has a powerful solvent action upon metallic oxides at a red heat. It is sometimes used as a flux.

**Zinc Chloride**,  $ZnCl_2$ , is obtained by dissolving zinc in hydrochloric acid. Its solution is the well-known soldering spirit. In the solid state it is used as a flux for aluminium.

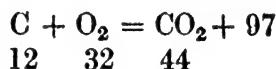
**Ammonium Chloride**, Sal-ammoniac,  $NH_4Cl$ , is a very volatile solid which is sometimes used as a flux for toughening gold and its alloys, and as a cover for zinc baths in the galvanizing process.

Other materials necessary for the work described in the following chapters will be referred to as they are required.

## CHAPTER II

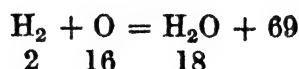
### FUEL

THE materials commonly used for the generation of heat are required in large quantities for various metallurgical and manufacturing processes. They are wood, peat, coal, oil, and their derivatives—charcoal, coke, coal gas, producer gas, water gas, and oil gas. All these fuels are very rich in carbon, and carbon in combination with hydrogen. Carbon and hydrogen are the heat-giving elements in natural fuels. Carbon when burnt completely gives out much heat, the chemical and heat changes being expressed by the equation :



That is, the complete combustion of 12 grams of carbon would develop sufficient heat to raise 97 kilograms of water through 1° C.

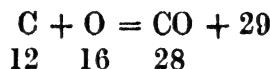
Similarly for hydrogen :



Or 2 grams of hydrogen completely burnt would raise 69 kilograms of water through 1° C.

But carbon may be partially burnt, in which case

carbon monoxide is formed, and the thermo-chemical equation is :



If, then, 28 parts of carbon monoxide are burnt,  $97 - 29 = 68$  kilograms of water would be raised through  $1^{\circ} \text{ C}$ . Thus the heat developing or calorific power of carbon monoxide is very high, and this compound is the most important in the composition of producer gas.

This chapter will deal with wood and coal, and their derivatives charcoal and coke. For further treatment of the subject special books must be consulted.

Charcoal is the purest of the solid carbon fuels, and is used for special work. Coal and coke, though impure, are more suited for ordinary purposes.

When either wood or coal is strongly heated out of contact with air, a large percentage of volatile liquid and gaseous matter is expelled. This, on cooling, separates into three distinct substances : (1) A watery liquid ; (2) an oily liquid or tar ; (3) a combustible gas. A black residue of charcoal or coke is left in the vessel used for heating. The proportion of this residue varies from about 20 per cent. in the case of wood to about 80 per cent. in that of deep coal. Coke from some coals is very friable and breaks up readily in the fingers ; from others it is well caked, dense, and hard.

Compounds of sulphur are found in all coals, so that sulphur must be regarded as a constant constituent. Also, when any of the solid fuels are burnt completely, a residue of earthy matter or ash is obtained, the amount of which varies considerably in the different varieties.

## 24 ELEMENTAL PRACTICAL METALLURGY

The general composition of coke, exclusive of the small quantity of volatile matter left in from the coking, is carbon 90 per cent., ash 7 per cent., moisture 2 per cent., and sulphur 1 per cent.

*Exp. 1.* Pack some splints of wood into a test-tube so as to occupy about half the length of the tube ; hold the open end in a stiff paper clip ; heat the part of the tube containing the wood in a Bunsen flame, holding the tube in a slanting position with the open end downwards ; place a piece of paper on the bench directly underneath the end of the tube, so that any liquid matter will drop on to it. Continue the heating until some liquid has been collected, then apply a light to the escaping gas, which will burn with a luminous flame. Allow the tube to cool, break it, and examine the black residue of charcoal.

The liquid matter will be seen to consist of water and tar. The former contains a number of substances in solution, such as acetic acid and wood spirit ; the latter is a very complex body ; while the gas consists mainly of hydrogen and marsh gas,  $\text{CH}_4$ .

**Coking.** This term, used in a general sense, means the heating of any kind of solid fuel out of contact with air to such a temperature that all volatile matter is expelled, and a solid residue containing fixed carbon and ash-forming substances only is left. The vessel for the heating cannot be completely closed as a way must be left for the escape of the volatile matter ; but it may be so arranged that very little air enters during the operation. To get a fair idea of the nature of the coke, the raw fuel should be reduced to a coarse powder before coking.

There are three varieties of coal : (1) Lignite ; (2) common coal ; (3) anthracite. Coal in all its qualities and anthracite are found in Great Britain ; lignite is a continental coal. Wood, lignite, and anthra-

cite give a powdery coke. That is, there is no tendency to softening and clotting of the particles of the original powder. Coal gives a more or less coherent mass which is friable in the case of free-burning coals, but dense and hard from coking coals. Also the percentage of volatile matter varies considerably. Gas coals give upwards of 40 per cent., but good coking coals as low as 20 per cent., of liquids and gases.

*Exp. 2.* Nearly fill a clay crucible with wood sawdust; tip it out and weigh it to the nearest gram, adding or removing a little as required. Return the weighed dust to the crucible and lute on the lid with moist clay, leaving a space near the lip for the escape of gaseous matter. Place the crucible well down in a good fire, but so that the lip can be seen. Nearly close the furnace, and observe the escape of gas from the crucible, noting the length and duration of the flame. When the flame drops cover the crucible over with hot coke, close the furnace, and leave it for five minutes. Remove the crucible and allow it to get cold with the lid on. Knock off the lid; weigh and examine the residue. Reserve for further use.

*Exp. 3.* Weigh 20 grams of powdered anthracite, and follow out exactly the instructions for the last experiment. Weigh and examine the residue.

The sawdust gives a dry black powder, the percentage of which is easily obtained from the weight of charcoal  $\times 100 \div$  the weight of sawdust taken.

The anthracite appears to undergo very little change but loses weight. The percentage of residue is obtained by multiplying its weight by 5, since 20 grams are taken. It should be noted that while the percentage residue from wood is about 20, that of anthracite is nearly 90. This comparison makes clear the great concentration of carbon during the passage of wood into coal.

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*Exp. 4.* Take 20 grams of coarsely crushed coal and proceed exactly as in *exp. 2.* Weigh and examine the coke, and calculate its percentage. One hundred — per cent. of coke = per cent. volatile matter.

If two samples can be obtained, one a moderately free burning coal and the other a good coking variety, a comparison may be made; but ordinary furnace coal will usually furnish a fairly coherent coke.

**Ash in Wood, Coal, and Coke.** The inorganic matter in fuel, which is left as a residue on its complete combustion, can readily be determined. This forms the ash, the principal constituents of which are silica, alumina, lime, magnesia, and ferric oxide. The colour varies from white to red.

*Exp. 5.* Weigh a clean porcelain crucible which has been heated and allowed to cool to ensure its being dry. Nearly fill the crucible with small pieces of wood, and weigh again. The difference gives the weight of the wood taken. Place the crucible on a roasting dish in a hot muffle and leave it until the wood is completely burnt. Weigh the crucible when cold, and calculate the percentage of ash.

### EXAMPLE

Wgt. of crucible + wood	28.065.	Wgt. of crucible + ash	21.095
" "	21.065	" "	21.065
	<hr/>		<hr/>
"	" 7.000	"	" .030
Percentage of ash = $\frac{0.03}{7.00} \times 100 = .43.$			

*Exp. 6.* Repeat, using 2 grams of finely powdered coal. The burning is accelerated if the mass is stirred from time to time with a bent-iron wire, but the wire should be tapped on the edge of the crucible before lifting it out, to prevent loss.

*Exp. 7.* Repeat, using 2 grams of finely powdered coke. Stir repeatedly, for coke is more difficult to burn than coal. See that the residue is quite "dead" before weighing. If time permits, the crucible should be put back in the muffle for 15

minutes, and reweighed when cold. If no further loss in weight occurs, the burning is complete.

As 2 grams are taken, the percentage of ash is given by weight of ash  $\times$  50.

The amount of ash in coal varies considerably, say, from 3 to 30 per cent., but is usually under 10 per cent. The percentage of ash in coke is, of course, higher than in the parent coal after washing, but should not exceed 7 per cent.—that is, coking coal should be low in ash.

**Moisture in Coal and Coke.** Free water is present in the raw material as it comes from the mine, but some is absorbed from the atmosphere, and in the case of coke during the manufacture.

*Exp. 8.* Weigh out 5 grams of finely powdered coal on glazed paper. Transfer it to a dry watch-glass; place another glass of the same size underneath, and weigh the whole. Place in a drying oven at 100° C, and leave for 45 minutes. Take out and cover the dried coal with the bottom glass, and allow to get cold. Weigh and note the loss in weight. Put the top glass back into its old position, return to the oven for 15 minutes. Reweigh when cold. If no further decrease in weight is obtained, the determination is finished. If further decrease, repeat the re-heating till the weight is constant.

Loss of weight  $\times$  20 = percentage of moisture.

*Exp. 9.* Repeat the experiment with 2 grams of finely powdered coke.

**Sulphur in Coal and Coke.** Sulphur occurs in coal as iron pyrites  $FeS_2$ , as calcium sulphate  $CaSO_4$ , and as organic sulphur compounds. It is only partially removed in the coking process, and, as its presence is always more or less objectionable in manufacturing processes, a knowledge of the amount present is useful.

*Exp. 10.* Weigh out 2 grams of finely powdered coal, and transfer it to a large porcelain crucible. Take 3 grams of

## 28 ELEMENTARY PRACTICAL METALLURGY

magnesia and 1 gram of sodium carbonate; well mix them, and add the mixture a little at a time to the coal mixing with a thin glass rod after each addition. Cover the whole with 1 gram of magnesia. Transfer the crucible to a roasting dish, and put it in the front part of a moderately hot muffle. After a few minutes push it back into a hotter part; close the muffle, and leave it for an hour. Take out the crucible, and when the redness has disappeared, stir up the mass with an iron wire to see if the coal is completely burnt. This is indicated by the absence of black particles. If not, put the crucible back for 15 minutes.

Transfer the properly burnt mixture to a clean beaker. Measure 50 c.c. of distilled water, and wash out the crucible with it, adding the washings to the beaker. Add 20 c.c. of strong hydrochloric acid, and a few drops of bromine water. Cover the beaker with a clock-glass, and put it on the hot plate to boil. Continue the boiling for 10 minutes; filter into a clean beaker, and well wash the residue with hot water. Neglect the residue on the filter. Boil the filtrate on gauze over the Bunsen flame, and when boiling add 10 c.c. of barium chloride solution; continue the boiling for 2 minutes, and allow to stand for a short time for the precipitate to settle. Filter and well wash the precipitate on the filter; place the filter in a cone on the hot plate to dry. Neglect the filtrate. Heat a porcelain crucible and lid on a clay triangle over the Bunsen flame, and when cold weigh both together. Transfer the dry precipitate from the paper to the crucible as far as possible, and burn the paper on the lid. When it is completely burnt add the ash to the crucible, and heat the whole to a low red heat on the clay triangle. Put on the lid and when the crucible is quite cold, weigh it. Calculate the percentage of sulphur.

### EXAMPLE

Weight of crucible + precipitate + ash	=	23.674
"	"	= 23.527
"	"	= .147
"	"	= .001
"	"	= .146

Weight of sulphur in precipitate

$$= \frac{S}{\text{BaSO}_4} \times \text{weight of precipitate}$$

Weight of sulphur in precipitate

$$= \frac{32}{233} \times .146 = .0209$$

Then percentage of sulphur in coal

$$= \text{weight of S} \times 50 = .0209 \times 50 = 1.04 \text{ per cent.}$$

This is so because the precipitate is barium sulphate and contains all the sulphur originally present in the coal. Exactly the same procedure is followed for sulphur in coke.

**The Calorific Power of Fuel.** By this is meant the total quantity of heat given out by the complete combustion of a given weight of the fuel. The simple plan is to burn the fuel and absorb the heat in water. The number of heat units given out is obtained by multiplying the weight of water by its rise in temperature. The most accurate method of determining calorific power is by means of a calorimeter in which the fuel is burnt in compressed oxygen in a strong bomb. The bomb itself is immersed in a known weight of water contained in a protected vessel, so as to prevent loss of heat. When the temperature has been equalized, and the difference between the initial and final temperature of the water observed, the necessary data for calculating the calorific value are obtained. Any kind of fuel can be dealt with in this apparatus, but it requires considerable technical skill in its manipulation.

**The Thompson Calorimeter** is easy to work, but is only suitable for solid fuel. Its accuracy is not very great, but it can be used with confidence to determine the relative values of two or more samples of coal, etc.

The general form of the apparatus is shown in Fig. 17. *A* is a tall glass cylinder graduated to contain 2000 c.c. of water, up to the mark *B*. *C* is a cylindrical metal chamber open at the bottom, and fitted with a long metal tube *D* furnished with a tap at the top. There is a ring of holes round it at the bottom for the escape of gas. *E* is a stand with spring clips, over which the chamber can be pressed and kept in position. *F* is a metal cylinder open at the top to contain the charge of fuel and oxygen compounds for burning the same. The thermometer is not shown.

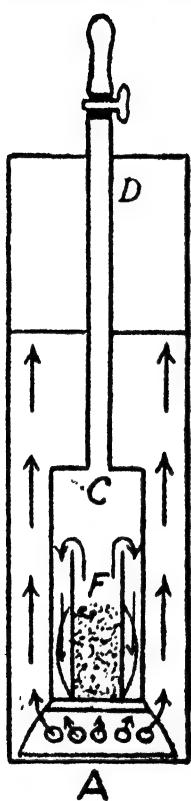


FIG. 17

The best results are obtained with ordinary coal, but at times even that is not entirely burnt, and its full value not obtained. Anthracite and coke are very difficult to burn in this apparatus, but by mixing with them a known proportion of wood charcoal the calorific value of which has been previously determined, a fairly good result may be obtained.

**The oxidizing agent** is a mixture of potassium nitrate  $KNO_3$ , 1 part, and potassium chlorate  $KClO_3$ , 3 parts. The salts are carefully dried and finely powdered before mixing. The fuel should also be finely powdered and dry. Care must be taken to have all the materials thoroughly dry, for success is largely dependent upon this. The **fuse** for igniting the mixture is prepared by soaking lamp-cotton in a strong solution of potassium nitrate, and drying it on the hot

plate. The salts and fuse are kept in stoppered bottles to prevent absorption of moisture.

*Exp. 11.* Stand the glass cylinder on the working bench, fill it up to the mark with water, and suspend a thermometer in it. Weigh out two grams of dry coal, 5 grams of potassium nitrate and 15 grams of potassium chlorate, all thoroughly dry. Mix them by grinding together in a porcelain mortar. Transfer the mixture to the ignition cylinder, which must be dry, and tamp it down with the wooden end of the pestle. Press into the top of the mixture one or two strands of the fuse cotton, and place the cylinder in the stand. Take the temperature of the water; ignite the fuse and quickly press the metal chamber over the springs. Hold the whole in the top of the glass cylinder until gas begins to escape, then plunge it into the water, and hold it in position. The combustion is very rapid, and the pressure of the escaping gases prevents water from entering the chamber. When the action is finished turn on the tap to flood the chamber and bring the whole to the same temperature as the water. Take the temperature of the water.

Assuming that 2000 c.c. of water weigh 2000 grams, which is sufficiently accurate for the purpose, then since 2 grams of coal are taken, the number of heat units given out by the combustion of 1 gram is found by multiplying the rise in temperature by 1000.

$$\therefore \text{C.P.} = (t_1^\circ - t^\circ) 1000$$

where  $t^\circ$  is the initial and  $t_1^\circ$  the final temperature of the water in the calorimeter. But it has been shown that the loss of heat, due to various causes, such as radiation from the apparatus, heat carried off by the escaping gases, and incomplete combustion amounts to 10 per cent. of the whole. Therefore one-tenth of the value found above must be added to it to obtain the correct value. An example will make this clear.

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Weight of coal burnt	=	2 grams
"      water heated	=	2000 "
Final temperature of water	=	19.5° C.
Initial    "            "	=	12.3° C.

$$\text{Increase in Temperature} = 7.2^\circ \text{ C.}$$

$$\therefore \text{C.P.} = 7.2 \times 1000 + 7.2 \times 100 = 7920 \text{ heat units.}$$

But some users prefer to have the statement in steam units, that is the number of units of water at 100° C. converted into steam at the same temperature. Now the latent heat of steam is 537 units: that is the quantity of heat required to convert unit weight of water at 100° C. to steam at the same temperature would raise 537 units of water through 1° C.

$$\therefore \text{C.P.} = \frac{7920}{537} = 14.75 \text{ steam units.}$$

In one form of the apparatus the glass vessel is graduated to contain 29010 grains of water up to the mark. In this case 30 grains of the fuel and 300 grains of the oxidizing mixture are required. The Fahrenheit scale of temperature is used, and since the latent heat of steam on the Fahrenheit scale is 967 units, and  $967 \times 30 = 29010$ , it follows that the calorific power in steam units is given directly by the increase in temperature in degrees F. Thus, in a case where the increase is from 53.6° F. to 66.6° F., the calorific power  $= 13 + 1.3 = 14.3$  steam units.

The chief value of the apparatus lies in the fact that it is easily manipulated, and that it gives sufficiently reliable data to distinguish between the relative values of different samples of fuel.

Exp. 13. Make a thorough mixture of two measures of raw clay and one measure of burnt clay, and weigh out the following quantities :

- (1) 40 grams of the mixture.
- (2) 36 grams of the mixture and 4 grams of lime.
- (3) 38 grams of the mixture and 2 grams of sodium carbonate.
- (4) 36 grams of the mixture and 4 grams of oxide of iron.

Transfer the clay and lime to a mortar, thoroughly mix by grinding with the pestle, and empty the mixture on to a piece of stiff paper. Repeat with the clay and carbonate, and the clay and oxide. Place the clay mixture (1) on a piece of paper, moisten it with a little water, and knead it into a stiff paste. It should not be wet enough to stick to the hands when rolled between them. A good plan to avoid getting the mixture too wet, is to moisten part of it and then to work the remainder in. Divide the plastic mass into two parts, and make two four-sided pyramids. This is easily done with the aid of a spatula.

Mark the bottom of each thus: I. Repeat with the other mixtures, and mark them II, III, IV respectively. Place the pyramids on a hot plate to dry; then transfer them to a cold muffle, start the muffle, and allow it to get to a bright red heat. Turn off the gas and let the contents remain until the muffle is cold.

Small crucibles may be hand-made, and for this purpose the mould and tools shown in Fig. 18 are used. The block A has a cavity in the middle with a groove running round it near the top. The brass mould B has two

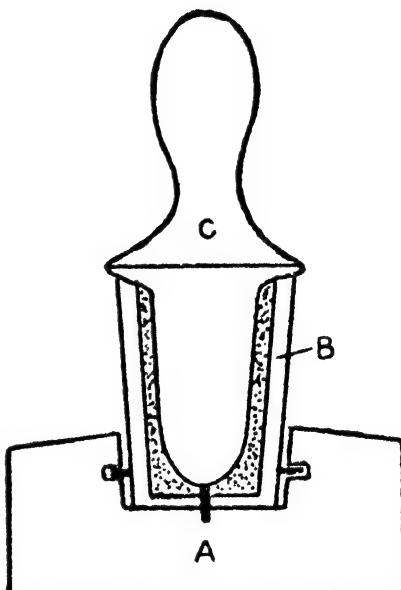


FIG. 18

## 36 ELEMENTARY PRACTICAL METALLURGY

pegs near the bottom which fit into the groove and keep the mould in position. The shaping tool C has a peg at the bottom which fits into a hole in the stand and keeps the tool central. The finishing tool has no peg, and is used to press a small piece of clay into the hole in the crucible made by the peg. The finished crucible is pushed out of the mould from the bottom.

*Exp. 14.* Work up the remainder of the mixture of raw and burnt clay into a very stiff paste, and thoroughly knead it. Mould three or four crucibles and lids, and make one disc about 2 in. by  $\frac{1}{2}$  in. thick. Keep the mould and shaping tools well oiled while moulding. Dry the crucibles, etc., and bake them along with the pyramids.

It is a good plan, when the lessons are of short duration, to leave the baking to be carried out in the interval between two lessons.

*Exp. 15.* Mix together one part of raw clay and one part of crushed and sifted coke, or powdered graphite; knead into a stiff paste; and mould one or two crucibles. Dry on the hot plate, and, while warm, paint the surface of the crucibles with a strong solution of borax. Bake in the muffle.

The borax will melt and form a thin glaze to protect the carbon from too rapid oxidation.

*Exp. 16.* To test the pyramids already prepared, place the four separately in clean crucibles with the apex downwards, and well cover No. IV with powdered charcoal. Lute on the lids with moist clay, and put the four crucibles one in each corner of the fire; cover well with coke, and close the furnace for 60 minutes. Care should be taken to have a good fire or the test will be unsatisfactory. Remove the crucibles from the fire, knock off the lids and examine the contents.

No. I, being a good clay mixture, should show no signs of fusion on the edges. Nos. II and III will show

signs of fusion, which is more pronounced in the case of No. III. This is due to the fluxing action of the oxides introduced, of which the soda has the greater action, although present in smaller quantity. With No. IV the action is very marked as the oxide of iron is reduced by the carbon introduced. It is quite clear from these tests that the smaller the percentage of metallic oxides other than alumina present in a fire-clay the more refractory it will be.

*Exp. 17.* Test a crucible for resistance to corrosion. Put 10 grams of red lead into the soundest of the crucibles made in (14). Put one of the round plates in a hot muffle, and place the crucible upon it. Note the time it takes the lead oxide to appear on the outer surface of the crucible. For two crucibles made under the same conditions, but from different clay mixtures, the time test is very instructive.

In testing an unknown clay, a portion of it should be heated to a red heat on a clay dish in a muffle to produce the "burnt" clay for the mixture, and this mixed with the raw clay in the proportions given above. Pyramids are then made from this mixture, and from a mixture of a standard clay, dried and burnt as described. The pyramids are put into crucibles, the lids luted on, and the crucibles clayed side by side, but not too close together, on a piece of fire-brick. The fire having burnt down, the bottom is cleared and the crucibles with the brick are lowered on to the fire-bars. The fire is then made up and kept well going for two hours. The pots are removed, and the test pyramid is compared with the standard.

**Casting Sands.** As already remarked, pure silica sand has no binding properties, but some sands contain sufficient clay "bond" to enable their particles to hold

## 38 ELEMENTARY PRACTICAL METALLURGY

together when the mass is moistened with water and pressed. These are the well-known casting sands, and may contain as much as 10 per cent. of clay matter mixed with the free silica.

*Exp. 18.* Mix together 9 grams of silica sand and 1 gram of dry fire-clay; transfer to a porcelain dish, add a little water, and thoroughly mix with the finger. Pour the supernatant water, which will be discoloured owing to the clay particles in suspension, into a beaker and repeat the washing and decantations until the washings are quite clear. Drain away as much water as possible; and place the dish on the hot plate to dry. The dried sand should weigh 9 grams if the washing has been carefully done. The clay will slowly settle to the bottom of the beaker.

*Exp. 19.* Repeat the above with 10 grams of casting sand. The loss  $\times 10$  will give the percentage of clay bond.

*Exp. 20.* Moisten some of the casting sand so that, when a portion is gripped in the hand, it will cling together. Make a core about 9 in. long and 1 in. in cross section by ramming the moist sand between two 1 in. bars placed side by side on a flat board and held in position. Remove the bars and push the core gradually over the edge of the board and at right angles to it. When the weight of the overhanging portion is sufficient to overcome the cementing action of the clay matter in the sand, the piece will drop off.

This is a good method of roughly comparing the binding properties of two sands. A further experiment may be made by mixing 10 per cent. of clay with the sand and proceeding as before. The length supported should be considerably increased by the introduction of the clay.

*Exp. 21.* Weigh out 9.5 grams of clean silica sand and .5 gram of red oxide of iron; well mix the two and transfer the mixture to a clean crucible; lute on the lid. Make up a similar crucible with 10 grams of the sand alone. Place the two crucibles on the fire; well cover them with coke, and heat strongly for 45

minutes. Take the crucibles from the fire and, when cold, remove the lids and examine the contents.

The sand alone is quite unchanged, but the portion mixed with the iron oxide is fritted and forms a fairly compact mass. This property of impure sands is often utilized in the formation of furnace beds, as they hold together better than purer materials, and are sufficiently refractory for the purpose.

**Slags.** The slags met with in metallurgical processes are for the most part complex silicates. That is, they are combinations of silica with various metallic oxides. When they contain only small percentages of the metals under treatment, and may go to the tip, they are to be regarded as true slags; but when they are produced in refining processes and contain much of the metals being operated upon, they are better known as scoria or cinders, and undergo further treatment for the extraction of the metals.

Of the single silicates, those containing sodium, potassium, and lead oxides are the most fusible; those of copper and iron take an intermediate place; while those of lime, magnesia, and alumina are the most infusible. But the fusibility also depends upon the proportion between the base and the silica. There are several types of silicates, but those mostly formed as slags are the monosilicates ( $2MO \cdot SiO_2$ ), and the bisilicates ( $MO \cdot SiO_2$ ). For the same base, the mono- is more fusible than the bisilicate.

A complex silicate is, as a rule, more fusible than the single silicate in its composition having the higher fusing-point. Thus the introduction of another base is commonly resorted to for slagging-off a refractory silicate.

## 40 ELEMENTARY PRACTICAL METALLURGY

*Exp. 22.* Weigh out 10 grams of silica sand and 18 grams of sodium carbonate ; well mix in a mortar ; transfer the mixture to a clay crucible, and melt in a sharp fire. Pour into a round mould. If the crucible is clean the glass will be colourless and transparent. It has a composition corresponding to  $\text{Na}_2\text{O} \cdot \text{SiO}_2$ , i.e. bisilicate.

*Exp. 23.* Weigh out 20 grams of litharge and 5.4 grams of silica ; mix, and melt down in the pot used for the sodium silicate. Pour into a round mould. The colour of the glass is due to the impurities, and varies from light to dark amber. It is also a bisilicate,  $\text{PbO} \cdot \text{SiO}_2$ .

*Exp. 24.* Repeat, using 20 grams of litharge and 2.7 grams of silica. The product is the monosilicate,  $2\text{PbO} \cdot \text{SiO}_2$ .

*Exp. 25.* Crush part of the lead glass obtained in the last experiment ; weigh 20 grams of it and 4.5 grams of small pieces of thin sheet iron ; mix, and melt in the same crucible. The fire must be hot enough to melt the iron sulphide formed in the reaction. Run the molten mass into a round mould, and, when cold, detach the slag and weigh the button of lead.

The last experiment is useful in showing how one metal may replace another in silicates. The change is given by  $2\text{PbO} \cdot \text{SiO}_2 + 2\text{Fe} \rightarrow 2\text{FeO} \cdot \text{SiO}_2 + 2\text{Pb}$ . Thus the weight of iron required, or the weight of lead liberated, is easily calculated.

*Exp. 26.* Weigh out 10 grams of black oxide of iron (hammer scale), .3 gram of charcoal, 2 grams of sodium carbonate, and 5 grams of silica. Grind the black oxide and the charcoal together, then mix in the carbonate and sand. Transfer the mixture to a clay crucible, and heat very strongly with the lid on for 20 minutes. When fluid, pour the melt into a round mould. The product is a monosilicate,  $2\text{FeO} \cdot \text{SiO}_2$ , of the same general composition as tap cinder, but purer. The soda is added to assist the fusion.

*Exp. 27.* Weigh out 10 grams of black oxide of iron, .3 gram of charcoal, 4 grams of lime and 8 grams of sand. Grind the black oxide and charcoal together, mix in the lime and sand, and heat the mixture very strongly in a clay crucible with the lid on. A rather higher temperature than that obtained in the

last experiment is required. Pour into a round mould. The product appromixates closely to the slag obtained from an ironfounder's cupola.

Tap cinder is a product of the puddling process, and although very impure, consists mainly of the monosilicate. The small quantity of charcoal is introduced to reduce the ferric oxide in the scale to ferrous oxide, as the higher oxide does not combine with silica. Cupola slag is not so rich in oxide of iron as tap cinder. The lime is put into the cupola charge to flux the silica and to prevent too great a waste of iron.

*Exp. 28.* Weigh out 10 grams of silica, 5 grams of lime, and 3 grams of alumina. Mix well, and transfer the mixture to a carbon-lined (brasqued) crucible; lute on the lid, and put the crucible into a good fire. Make up the fire well over the pot, and leave it for 45 minutes, or longer if convenient. Allow the pot to cool before removing the lid. The product is a bisilicate of lime and alumina.

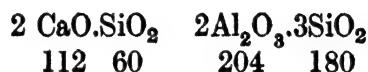
*Exp. 29.* Mix together 5 grams of silica, 6 grams of lime, and 2 grams of alumina. Repeat the last experiment. The product is a monosilicate of lime and alumina.

The first of these double silicates,  $3\text{CaO}\text{SiO}_2\cdot\text{Al}_2\text{O}_3\cdot 3\text{SiO}_2$ , forms the principal part of the slag produced in an iron-smelting blast furnace working with a cold blast; while the second is the result of hot blast working. Its formula is  $6(2\text{CaO}\cdot\text{SiO}_2)\cdot 2\text{Al}_2\text{O}_3\cdot 3\text{SiO}_2$ . As produced above, they are dark coloured owing to the presence of the charcoal brasque, which is used to prevent the slag from sticking to the pot. The temperature of the furnace is not usually high enough to admit of the slags being poured. The percentage composition of these slags should be worked out from the formula given, and the atomic weights on p. 15.

The fluxing action of soda, lead oxide, iron oxide,

## 42 ELEMENTARY PRACTICAL METALLURGY

and lime is clearly shown by the above experiments; but it would be useful, if time permits, to take the double monosilicate containing  $2\text{CaO} \cdot \text{SiO}_2$  and  $2\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$  and endeavour to form these silicates separately. The proportional quantities are easily calculated from the formulæ :



If convenient quantities of the mixtures are heated as strongly as possible for more than an hour in brasqued crucibles, their comparative infusibility when separate will be demonstrated.

## CHAPTER IV

### FORMATION AND REDUCTION OF OXIDES

THE atmosphere contains four principal gases, oxygen, nitrogen, carbon dioxide, and water vapour. Of these, oxygen forms about one-fifth and nitrogen four-fifths of the bulk. Carbon dioxide and water vapour, though necessary to a good working atmosphere, are present in very much smaller quantities. All the common metals, except gold, silver, and platinum, are oxidized when heated in air. That is, they absorb oxygen, increase in weight, and are converted into oxides. The apparatus shown in Fig. 19 is to be used for the following experiment :

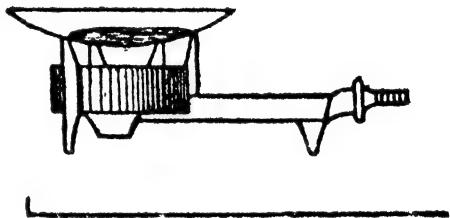


FIG. 19

*Exp. 30.* Weigh out 25 grams of lead and heat it in a clean iron pan over a good Bunsen flame. Keep the molten metal well stirred, and see that the yellow powder formed does not get hot enough to melt. When the metal has apparently all disappeared, allow the pan to cool, and weigh the powder on a counterpoised paper. There should be a distinct increase in weight.

*Exp. 31.* Weigh 5 grams of tin and 5 grams of lead. Melt the metals together on the pan and keep well stirred as described above. The oxidation will be much more rapid with the alloy

## 44 ELEMENTARY PRACTICAL METALLURGY

than with lead alone, and care is required to avoid loss. When finished, allow to cool and weigh the powder.

If the powder is ground in a porcelain mortar and washed by repeated additions of small quantities of water with stirring, and the muddy liquid is poured into a large porcelain basin after each addition, the oxides may be separated from the unchanged metal. The clear liquid, after settling, may be poured off, and the solid residue dried on the hot plate. The yellow lead oxide so obtained is commonly known as "massicot" and may be "coloured" by long-continued heating at a rather lower temperature than that used in its preparation. The product is red lead  $Pb_3O_4$ , as distinguished from massicot  $PbO$ , and is formed by the absorption of more oxygen from the air. This excess oxygen is however expelled at a higher temperature. The mixture of lead-tin oxides is used as a polishing powder for needles, etc.

*Exp. 32.* Weigh out 5 grams of tin, transfer the metal to a roasting dish, and place in a muffle which is at a moderate red heat. Keep the contents of the dish well stirred from time to time, and when the metal is all oxidized remove the dish; allow it to cool and weigh the white powder.

It is seldom that the lead is completely oxidized, but with care a quantitative result is obtained with tin. The powder is tin oxide,  $SnO_2$ . The weight of oxide is given by weight of tin  $\times \frac{151}{119}$ .

*Exp. 33.* Weigh accurately a strip of copper about 3 grams; bend it so that it will stand on end on a roasting dish. Put the dish in a moderately hot muffle, and leave for an hour. Then tip the strip into a porcelain mortar and reduce it to powder. Whether the whole of the metal has disappeared or not, note the colour of the powder and return it to the dish. Use a camel-hair brush to remove the last portions from the mortar. Put

## FORMATION AND REDUCTION OF OXIDES 45

the dish back in the muffle for fifteen minutes, but do not let the temperature rise too high or the oxide will become pasty and stick to the dish. Allow to cool and then weigh. Repeat the grinding and heating if necessary.

Copper forms the red oxide first, and this changes to the black oxide by further absorption of oxygen. If the oxidation is complete, weight of oxide = weight of metal  $\times 1.25$ .

*Exp. 34.* Repeat, with a piece of iron wire which has been rolled into a thin ribbon, in place of the copper.

$$\text{Weight of oxide} = \text{weight of metal} \times 1.38.$$

The rate at which different metals absorb oxygen varies considerably. Thus iron oxidizes rapidly, nickel slowly, and silver not at all.

*Exp. 35.* Take two pieces of sheet iron and sheet nickel of exactly the same area; weigh them separately, and place them side by side on a roasting dish. Put the dish in the muffle and leave it for 30 minutes. Remove and, when cold, reweigh. Compare the increase in weight of the separate pieces.

The above experiments illustrate the direct oxidation of metals, but oxides may be formed from salts of the metals by strongly heating them.

*Exp. 36.* Weigh out 20 grams of powdered copper sulphate; transfer it to a roasting dish, and place in a moderately hot muffle. Leave in the muffle for half an hour, stirring the powder from time to time with an iron stirrer. Take the dish out and note if sulphur fumes are still escaping, and if the residue is black all through. Return if not quite finished. Weigh the black powder, and reserve for future use.

$$\text{Weight of oxide} = \text{weight of salt} \times 1.319.$$

*Exp. 37.* Weigh 20 grams of powdered ferrous sulphate and repeat. If the temperature is not too high the colour of the oxide (rouge) will be a fine medium red.

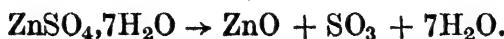
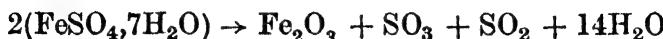
$$\text{Weight of oxide} = \text{weight of salt} \times 1.29.$$

*Exp. 38.* Weigh 20 grams of zinc sulphate and repeat.

$$\text{Weight of oxide} = \text{weight of salt} \times 1.32.$$

## 46 ELEMENTARY PRACTICAL METALLURGY

The reactions taking place in the above experiments are shown by the equations :



Carbonates and nitrates are also decomposed, leaving residues of oxides.

*Exp. 39.* Weigh a porcelain crucible and weigh into it about 2 grams of lead carbonate (white lead). Place the crucible on a clay triangle over a Bunsen flame and heat carefully until the mass is yellow all through. Weigh when cold and note the loss in weight due to the evolution of carbon dioxide. The residue is PbO.

*Exp. 40.* Repeat with 2 grams of cobalt nitrate. The residue is black cobalt oxide. Note the red fumes of nitrogen oxides given off during the heating.

**Reduction of Oxides** by carbon with separation of their metals is effected more or less readily, and depends upon the temperature employed. Thus, while lead oxide is reduced at a low red heat, tin oxide requires a bright red one for its reduction, and alumina is reduced only at the temperature of the electric furnace. A thorough mixture of the oxide and carbon should be effected before proceeding to the reduction.

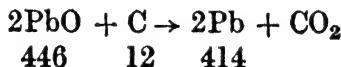
*Exp. 41.* Weigh out 20 grams of litharge, PbO, and mix with it .6 gram of powdered charcoal. Transfer the mixture to a small clay crucible, and heat it to low redness for a few minutes ; then place in a hotter part of the fire until the mass melts, and settles down in the bottom of the pot. Pour the melt into a round mould, and when the button has cooled detach the small quantity of slag usually formed, and weigh the metal button.

*Exp. 42.* Repeat, but use 1.5 grams of charcoal in place of .6. Note that the mass does not fuse properly, and that the metal

is for the most part in shots, distributed through the excess of charcoal. This excess may be removed by washing the product in a porcelain basin when the light charcoal is washed away.

*Exp. 43.* Repeat, using 3 of charcoal. Melt the charge rapidly, and pour as soon as thoroughly molten. A metal button will be obtained in a shell of unchanged litharge.

The chemical change is expressed by the equation :



From the formula weights, it is seen that the possible weight of lead to be obtained from 20 grams of the oxide

is given by  $\frac{414 \times 20}{446} = 18.565$ .

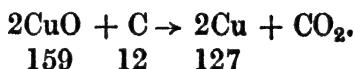
The experiments show that the nearest to this is obtained when the charcoal added is about that calculated from the equation, and that either an excess or deficiency gives a poorer result. But the possible weight cannot be obtained from a clay crucible as silicate is always formed. A nearer approximation would result from the use of a carbon crucible. An exact result is obtained if carefully dried litharge is reduced by coal gas in a hard glass-tube.

*Exp. 44.* Weigh out 5 grams of black oxide of copper and 1.7 grams of powdered charcoal. Well mix and heat strongly in a good fire. When the metal appears add a mixture of 10 grams of sodium carbonate and 2 grams of argol. Close the furnace for two or three minutes for the fluxes to melt, and allow the metal to gather together. Pour into a button mould; detach the slag, and weigh the button.

*Exp. 45.* Mix 5 grams of the copper oxide with 10 grams of sodium carbonate and 10 grams of argol and run down the mixture in the same crucible. Carbon, liberated from the argol on heating, does the work of the charcoal used in the first experiment. The button of copper obtained should have the same weight.

## 48 ELEMENTARY PRACTICAL METALLURGY

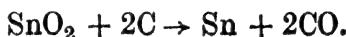
The change is given by the equation :



Calculate the weight of copper that should be obtained. Although a high temperature is used, it is not necessary for the actual reduction, but for fusing the reduced metal. The flux helps the copper to run together into a button.

*Exp. 46.* Well mix 5 grams of tin oxide and .9 gram of powdered coke. Transfer to clay crucible, and put well down in a good fire, so that it may be rapidly and strongly heated. The pot should be covered with a lid to keep out pieces of coke. The metal should appear in about 10 minutes. Remove the pot, tap it on the furnace top to shake the particles together, and tip the contents into a round mould. Weigh the main button together with any shots that may be found in the débris.

The change is given by



It is seen that the lower oxide of carbon is formed at the higher temperature required for the reduction. If the metal does not run together well a piece of potassium cyanide, the size of a small nut, dropped into the crucible will help the fusion. The cyanide itself is the best reducing agent for tin oxide, but as it is a strong poison much care is required in its use.

*Exp. 47.* Thoroughly mix 5 grams of zinc oxide and 1.5 grams of charcoal. Transfer the mixture to a clay crucible, and lute on the lid with moist clay, leaving a space near the lip quite clear. Heat strongly, and note the light-blue flame issuing from the crucible while the reduction is proceeding. Keep the pot in the fire for 5 minutes after the flame ceases. When cold, remove the lid and examine the contents of the pot. A small residue of charcoal is found.

## FORMATION AND REDUCTION OF OXIDES 49

The change is given by  $\text{ZnO} + \text{C} \rightarrow \text{Zn} + \text{CO}$ . As the zinc is volatile at the reduction temperature, it escapes from the crucible, and burns to oxide again.

*Exp. 48.* Weight out 5 grams of red oxide of iron and mix with it 3 grams of sodium carbonate and 2 grams of powdered glass. Transfer the mixture to a carbon-lined crucible, place a piece of charcoal on the top, and lute on the lid. Put the pot into a good fire and heat strongly for 45 minutes. Knock off the lid when the pot has cooled, and remove the button of slag and iron. Usually the reduced iron is in one piece, but if in shots, crush the slag and separate the shots by means of a magnet.

The iron so obtained is not pure, but contains 2 to 3 per cent. of carbon, and the button is readily broken by a blow from a hammer. The fractured surface is usually white and crystalline.

*Exp. 49.* Well mix 5 grams of nickel oxide and 5 grams of copper oxide with 15 grams of sodium carbonate and 15 grams of argol. Strongly heat the mixture in a clay crucible, and when in quiet fusion pour it into a round mould. Detach the button of metal from the slag, grip it in the vice, nick it with a chisel, and then break it. The fractured surface should be greyish-white in colour.

This experiment illustrates the production of an alloy by the reduction of its constituent metals from their mixed oxides. The alloy in this case is cupro-nickel and contains about 50 per cent. of each metal.

The combustible gases, carbon monoxide  $\text{CO}$ , marsh gas  $\text{CH}_4$ , hydrogen  $\text{H}_2$ , and gaseous hydrocarbons generally, will reduce a number of metallic oxides, when they are heated in contact with the gases. These reactions are easily illustrated by means of coal gas, 95 per cent. of which is a mixture of the gases named above.

*Exp. 50.* Fix in a clip a piece of combustion tubing drawn off at one end as shown in Fig. 20. Connect the thin end with the gas-supply by a rubber tube, and introduce about 2 grams of black copper oxide into the wide part of the tube. Place a Bunsen burner underneath, and heat the part containing the oxide. Turn on the gas at the same time, and ignite it at the open end to prevent the escape of unburnt gas into the air. Watch the gradual reddening of the black powder due to the appearance of metallic copper. Note that water condenses in the cool part of the tube. This results from the oxidation of

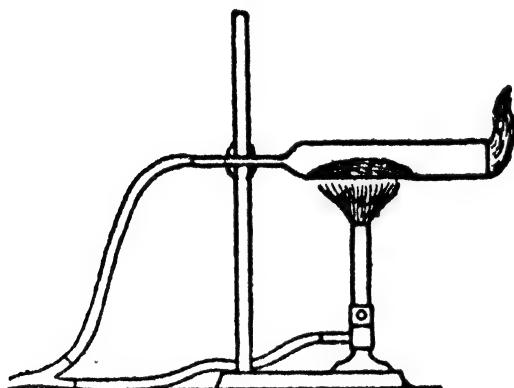


FIG. 20

the free hydrogen and of that in the hydrocarbons. When the oxide is all reduced take away the Bunsen flame, but allow the gas to continue burning at the end of the tube until the copper has cooled, or it will be reoxidized if air is let in while it is still hot. Remove the copper, and weigh it.

Compare the weight with the weight of oxide taken, and note the loss due to the removal of oxygen. Transfer the reduced copper to a porcelain crucible, and heat it on a clay triangle over the Bunsen flame. Note that it turns black, and regains its original weight.

Similar experiments may be made with lead and iron oxides respectively. In the case of the iron oxide, the reduced metal takes up carbon from the carbon monoxide and hydrocarbons, and so weighs more than it would do if this action were absent. If the reduced metal, which is in the form of a black powder, is heated in contact with air, it is changed back to the oxide; but the increase in weight is not so great as it would be if the powder were pure iron. The presence of the

carbon may be shown by heating some of the powder in a glass-tube, while a current of air is aspirated through it, and afterwards through lime water. The lime water is turned milky.

The formation of carbon dioxide when carbon or carbonaceous substances reduce oxides at a moderate temperature is readily shown.

*Exp. 51.* Mix together 3 grams of copper oxide and 1 gram of carbon ; heat the mixture in a test-tube fitted with a bung and delivery tube as shown in Fig. 21. Allow the gas given off to collect in the gas cylinder, and then add some clear lime water to it, and shake well. The lime water is turned milky, showing that carbon dioxide escaped from the tube in which the mixture was heated. Lead oxide may be used instead of copper oxide in this experiment.

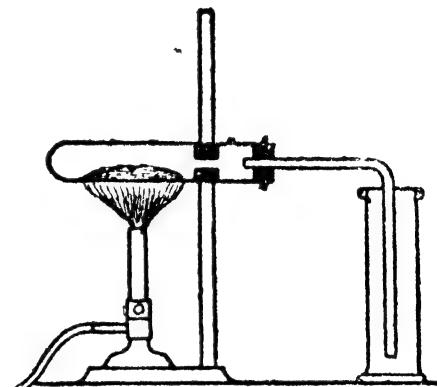
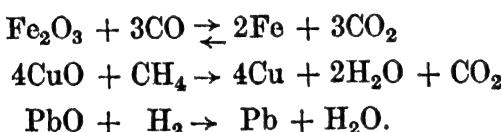


FIG. 21

The reactions taking place between the oxides and gases may be seen from the equations :



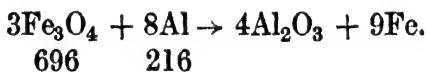
Another interesting reaction is that in which the oxygen is taken from an oxide by another metal. Thus the well-known *Thermit* process depends upon the reduction of iron oxide by aluminium, and the temperature generated by the reaction is so high that the reduced iron is in a perfectly fluid condition. *Thermit*

## 52 ELEMENTARY PRACTICAL METALLURGY

welding is the soldering of two pieces of iron together by the molten iron produced in the process. Quite a number of metallic oxides are reduced by aluminium, and valuable products, such as chromium and manganese, are obtained. The aluminium must be finely divided ; either filings or the granulated form obtained by vigorously stirring the molten metal in a crucible just as it is on the point of solidification. A very fine powder can be obtained in this way by sifting. But for small quantities it is less trouble to make the filings.

*Exp. 52.* Weigh out 20 grams of iron scale,  $\text{Fe}_3\text{O}_4$ , grind it to powder in a mortar, add to it 6.5 grams of finely divided aluminium and well mix. Put the mixture in a small clay crucible, and embed the crucible in casting sand contained in an iron mortar. Mix together 1 gram of barium peroxide and 1 gram aluminium. Make a depression in the top of the thermit mixture, and pour the peroxide mixture into it. Place a short piece of magnesium ribbon in it, and ignite the free end of the metal with a burning taper. Stand back a little until the reaction has abated, and then note the very high temperature generated in the crucible as shown by the white-hot mass it contains. Allow to cool, break the crucible and detach the button of metal from the slag. Test it with a file. It is fairly soft, nearly pure iron.

The reaction is expressed by the equation :



A little excess of aluminium is used over that found from the equation. The red oxide may replace the black oxide, but it does not work as well. Copper oxide is also readily reduced when mixed with three-tenths its weight of aluminium and fired as directed.

Iron alloys with chromium and similar metals may be prepared by this process.

## CHAPTER V

### FORMATION AND REDUCTION OF SULPHIDES

SULPHUR, like oxygen, unites directly with most of the metals, but a few, such as gold and platinum, do not so combine. Zinc sulphide is somewhat difficult to form by direct combination, but other common sulphides may be made the subjects of easy experiments. The readiness with which sulphur unites with metals varies considerably. Thus copper, nickel, and iron are in order, for if these metals are present in a mixture in which sulphides are being formed, sulphur will pass by preference to copper, then to nickel, and finally to iron.

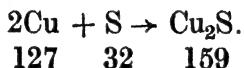
Sulphur, which is a yellow crystalline solid, melts at  $114^{\circ}$  C. to a thin amber-coloured liquid which thickens and becomes darker as the temperature rises. It boils at  $448^{\circ}$  C., giving off a deep red vapour. The commercial substance can be obtained either in lumps or as a powder.

*Exp. 53.* Put a clean clay crucible into a good fire to get hot. Take 25 grams of thin sheet copper, cut into small pieces ; mix with it 10 grams of powdered sulphur ; place the mixture in a cold iron shoot ; lift the hot crucible into a convenient position in the furnace, and shoot the mixture into it. Replace the crucible in its former position, put on a lid, and close the furnace. The mass will fuse to a clear liquid in a good fire. When fused, drop in one or two pieces of sulphur about the size of a small

## 54 ELEMENTARY PRACTICAL METALLURGY

nut ; replace the lid for a minute or two, and then pour the liquid into a button mould. Weigh the button when cold, and break it by tapping with a hammer. Reserve the sulphide for further use.

The reaction is expressed by the equation :



The compound formed is the stable sulphide of copper, as it will withstand a bright red heat without decomposition. The higher sulphide, CuS, is decomposed on heating into Cu<sub>2</sub>S and sulphur. As the molten sulphide wets the crucible, the weight obtained is short of that given by the equation ; but if a carbon crucible is used the full weight, 31.3 grams, is obtained from 25 grams of copper. If the sulphur used is insufficient, the excess copper will be found in the bottom of the button.

*Exp. 54.* Take 40 grams of granulated lead and 15 grams of powdered sulphur. Mix together and repeat the last experiment. Weigh, examine, and reserve the button for future use.

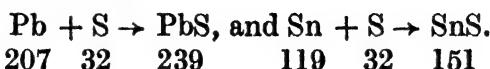
*Exp. 55.* Mix together 10 grams of granulated tin and 5 grams of powdered sulphur, and repeat the above.

Granulated tin or lead can be prepared by vigorously shaking the metal in a strong wooden box when just on the point of solidifying. The metal is melted and allowed to cool in the pot until just above its melting-point ; it is then poured into the box and shaken until it breaks up into granules. The product is then sifted through a 10-sieve, and that which does not pass through is returned to the pot for further treatment.

The sulphides prepared above are well defined crystalline solids, and illustrate clearly the radical

## FORMATION AND REDUCTION OF SULPHIDES 55

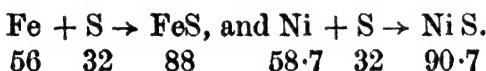
change in the properties of elements brought about by their chemical combination. The reactions for the last two are given by :



*Exp. 56.* Take 40 grams of thin sheet iron cut into pieces which can be loosely packed in an upright position in a clay crucible. Put the crucible and contents into a good fire, and when the metal is at a bright red heat, add with an iron shoot 10 grams of coarsely powdered sulphur. Note the increase in temperature due to the chemical action between the iron and the sulphur. Have two more similar quantities of sulphur ready and shoot them in at short intervals ; then replace the lid and close the furnace for two or three minutes. When the mass has run down, make the end of a thin iron poker red hot and stir the contents with it to see that the iron has completely melted. Drop in a piece of sulphur, close the furnace for a minute, and then pour the molten sulphide into a button mould. Weigh, fracture, examine and reserve the button of sulphide.

*Exp. 57.* Repeat the experiment with 10 grams of thin sheet nickel and about the same weight of sulphur, but do not stir with the iron poker, or the nickel sulphide will be contaminated with iron.

The changes are :



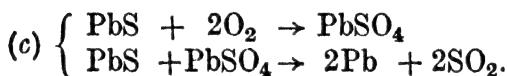
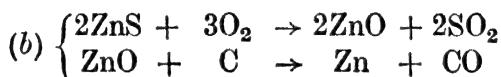
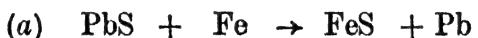
All the sulphide experiments may be made strictly quantitative if a carbon crucible is available, for the molten sulphides do not wet the crucible, and clean casts may be made.

*Exp. 58.* Melt 2 grams of fine silver in a carbon crucible, and drop in two or three pieces of sulphur. When the action is finished pour the sulphide into a round mould ; weigh and examine the button. It can be flattened somewhat by hammering, and when fractured shows a crystalline structure.

The change is :  $2\text{Ag} + \text{S} \rightarrow \text{Ag}_2\text{S}$

## Roasting and Reduction of Sulphides

There are three principal ways in which metals can be obtained from their sulphides: (a) By displacing the metal by means of another metal; (b) by roasting off the sulphur, and reducing the resulting oxide by carbon; (c) by roasting off part of the sulphur and reducing the unchanged sulphide by means of the oxide or sulphate formed during the partial roasting. The reactions are made clear by the equations:



*Exp. 59.* Mix together 20 grams of powdered lead sulphide and 5 grams of clean iron filings; transfer the mixture to a clay crucible and heat in a sharp fire until the mass is quite fluid; pour into a round mould. Hammer the cold button on the edge to displace the iron sulphide which adheres firmly to the lead and which, if hammered on the flat, is driven into the soft metal. Weigh the metal and compare the weight with that calculated from  $\text{PbS} + \text{Fe} \rightarrow \text{Pb} + \text{FeS}$ .

The experiment is more successful if 10–15 grams of sodium carbonate are added to the mixture before fusion, as the iron sulphide forms a more fluid slag with the carbonate.

Economy in the use of materials should be practised even when working on the small scale. This is kept in mind in giving the general instructions. As regards fluxes, if the melt is too thick to pour conveniently,

## FORMATION AND REDUCTION OF SULPHIDES 57

the addition of a little sodium carbonate or borax and further heating will improve the result.

*Exp. 60.* Repeat with 10 grams of antimony sulphide, 5 grams of iron filings and 10 grams of sodium carbonate. The heating must not be too prolonged as antimony is volatile ; also the metal does not separate as readily as lead on account of its lower density.

The change is given by :



**Roasting of Sulphides.** Generally, when sulphides are heated in contact with air, the sulphur burns off and oxides of the metals are formed. Also in most cases there is a direct absorption of oxygen by which sulphides are converted into sulphates. The latter, however, are for the most part decomposed at higher temperatures with liberation of oxides of sulphur, leaving the oxides of the metals. Lead sulphide is somewhat exceptional in this respect, and is almost completely converted into sulphate at muffle temperatures.

*Exp. 61.* Place 3 grams of finely powdered copper sulphide in a roasting dish, and heat carefully for 15 minutes over a round burner with constant stirring. See that the temperature does not rise sufficiently to cause the sulphide to soften and clot, and note that little or no sulphur dioxide is given off. Transfer from the dish to a clean mortar and grind ; brush the powder into a beaker ; add a little water, and boil for a minute or two. Filter, and if the filtrate is distinctly blue, evaporate it to a small bulk and set aside to crystallize, when crystals of copper sulphate will separate. If the colour of the filtrate is very faint, add ammonia solution. A deepening of the colour indicates that some of the sulphide has been sulphated and rendered soluble, which was to be proved.

The probable change is indicated by the equation :



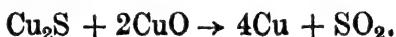
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which is supported by the fact that the residue on the filter is often distinctly red from the presence of cuprous oxide.

*Exp. 62.* Put 5 grams of powdered copper sulphide into a roasting dish ; place the dish in the front part of a moderately hot muffle ; stir continuously for the first few minutes, and gradually push the dish back into hotter parts of the muffle. Then stir occasionally, but see that the material does not clot. Continue the roasting as long as sulphur dioxide is given off, which is easily detected by withdrawing the dish and wafting the escaping gases towards the face. Weigh the product when cold. The weight should be the same, as  $\text{Cu}_2\text{S}$  becomes  $2\text{CuO}$  in the limit.

*Exp. 63.* Transfer the roasted mass to a mortar, and add to it the same weight of unchanged sulphide ; mix well by grinding. Now strongly heat the mixture in a clay crucible, when metallic copper is set free. As soon as the metal appears, add a little powdered charcoal, and continue the heating for a minute or two longer. Pour the contents of the crucible into a round mould and, when cold, weigh the button of coarse copper.

The change is given by :



The appearance of the button of copper may be improved by remelting it with 8 grams of sodium carbonate and 2 grams of argol in the same crucible.

*Exp. 64.* (a) Melt together 10 grams of iron sulphide and 5 grams of copper sulphide to produce a coarse matte containing about 27 per cent. of copper. The crucible used for preparing copper sulphide will serve for this melt. When the mass is thoroughly fluid pour into a round mould. Grind the matte in an iron mortar and pass it through a 30-sieve.

(b) Roast 10 grams of the powdered matte for 15 minutes, beginning at a moderate temperature and increasing it towards the end ; keep well stirred, especially at the beginning of the roasting. The change is not complete, as part only of the sulphur is removed and replaced by oxygen. The operation is known

as partial roasting, and the mixture contains oxides and unchanged sulphides.

(c) Grind the roasted matte in a mortar and mix with it 5 grams of fluor-spar, 5 grams of glass, and 15 grams of borax. Fuse the mixture in a clay crucible at a moderate red heat, and, when quite molten, pour it into a round mould. When cold, break away the slag from the button of fine matte. This must be done carefully, as the matte is brittle. Roughly crush the slag and pick out any shots of matte which may be mixed with it. The weight of the matte will vary with the extent of the roasting, and if this has gone too far a little coarse copper may be obtained.

(d) Reduce the matte to fine powder and roast it in the muffle with the precautions given above until all the sulphur is removed. Mix with it 5 grams of argol, 5 grams of sodium carbonate, and 3 grams of borax; transfer the mixture to the crucible used to obtain the fine matte, and fuse for about 10 minutes. When quite molten pour into a round mould, allow to cool, and separate the slag from the button of coarse copper. The metal should weigh about 2.5 grams.

The chief ore of copper is copper pyrites, which contains copper and iron sulphides ( $Cu_2S \cdot Fe_2S_3$ ) intermixed with earthy matter and other impurities. In extracting copper from this material, the metal is first concentrated in a matte, and the earthy matter, together with some of the iron and a little copper, is separated as a slag. Some iron sulphide is left in the matte, or too much copper would pass into the slag. As copper can be readily extracted from the ore on the small scale, as in the ordinary dry assay, the following experiments may be carried through.

The ore is supplied properly sampled, crushed to a fine powder, and sifted.

*Exp. 65.* Weigh out 25 grams of the ore, 25 grams of borax, 15 grams of glass, 15 grams of fluor-spar, and 10 grams of salt-petre. Well mix, and transfer the mixture to a large clay

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crucible, which should not be more than two-thirds full when the whole of the charge is in. Put well down in a good fire so that the sides of the crucible are uniformly surrounded with hot coke. Uniform heating is necessary, or the molten charge may boil over. As soon as the mass has settled down into tranquil fusion, pour it into a large round mould. Pinch the slag with the tongs as soon as it gets pasty, and so make a projection by which it can be lifted from the mould when it has cooled sufficiently. If time is of importance take out the button with the tongs, and pass it backwards and forwards through a stream of water from the tap, but leave enough heat in to dry it. Otherwise, let it cool in the mould. Break away the slag carefully, and remove the button of matte; examine the slag and pick out any shots of matte which may not have settled out. The slag may be thrown away.

The saltpetre is an oxidizing flux, and its principal function is to supply oxygen to iron and sulphur. Part of the iron is thus converted into oxide and passes into the slag. The remainder is present as sulphide with the copper sulphide in the matte. The earthy matter combines with the fluxes and forms the main bulk of the slag. The copper is thus concentrated with some iron in the matte, which is a mixture of sulphides of the two metals.

*Exp. 66.* Weigh the matte and grind it to a fine powder in the mortar. Wrap a duster round the handle of the pestle, and cover the mortar with it to avoid loss of material while grinding. Transfer the powder to a clean roasting dish, and roast it "sweet." That is, until the odour of sulphur dioxide can no longer be detected on wafting the hot gases from the dish towards the nose. This will take from 30 to 40 minutes, according to the quantity to be roasted. Remove the dish and allow it to cool.

The copper and iron are both converted into oxides, and the sulphur removed as sulphur dioxide. The powder should not be allowed to clot; this can be prevented by keeping the dish in the front of the muffle

for the first 10 minutes, and then gradually moving it back into the hotter parts.

*Exp. 67.* Grind the roasted matte if necessary, and mix it with 5 grams of sodium carbonate, 6 grams of tartar, and 5 grams of borax. Transfer the mixture to a clay crucible, and melt it in a good fire. The final temperature must be high enough to melt the reduced copper. Pour into a round mould, and when cold break away the slag to obtain the button of coarse copper.

The tartar supplies the carbon to reduce the copper oxide, and the fluxes generally absorb the iron oxide into the slag. The copper is usually impure from the presence of small quantities of other metals and sulphur. These can be nearly all removed and tough copper obtained by a refining process.

*Exp. 68.* Put the crucible used in the last experiment back into the fire, and when it is white hot, drop in the button of coarse copper. Nearly close the furnace, and watch when the copper melts and forms a globule, throwing off a greenish light. Then shoot in 5 grams of refining flux, close the furnace for one minute, and quickly pour into a round mould. Detach the button from the slag, and weigh it. It should flatten readily.

The slag from the coarse copper, and the refinery slag, both contain a small amount of copper. They may be treated together for its extraction.

*Exp. 69.* Roughly crush the slags obtained in the last two experiments. Mix the powdered slags with 5 grams of sodium carbonate, 5 grams of tartar and 1 gram of charcoal. Melt the mixture in the crucible used for refining, and, when tranquil, pour into a round mould. Allow to cool, crush in a mortar, and pick out any small shots of copper. Add them to the main button.

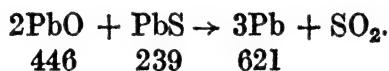
As 25 grams of the ore were taken, the weight of the copper multiplied by 4 gives the percentage of the

metal present. The processes described above are similar to those constituting the Cornish Dry Assay.

**Reduction of Lead Sulphide.** The principal ore of lead, galena, contains the sulphide. The ores from different districts vary very much as to their content of lead. The pure sulphide contains 86.6 per cent. of lead, and some of the rich ores contain as much as 80 per cent. of the metal. So that either the artificial sulphide or the ore may be used for the following experiments.

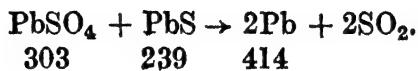
*Exp. 70.* Well mix 20 grams of litharge with 15 grams of powdered lead sulphide. Transfer the mixture to a clean clay crucible, and heat gradually to a good red heat, keeping the mass in a pasty state for some minutes. When the mixture is thoroughly molten and tranquil, pour it into a round mould. Leave in the mould until the metal is quite set. Hammer the button on the edge to break away the slag, and weigh the lead.

As an excess of lead sulphide is present in the mixture the reduced metal is usually hard, and the slag is rich in lead. The reaction is expressed by the equation :



Therefore the theoretical weight of sulphide to react with 20 grams of litharge is  $\frac{239}{446} \times 20 = 10.72$  grams ; but an excess gives a better practical result.

Lead sulphate also reacts with the sulphide, and lead is set free as shown by the equation :



But the result obtained by using the prepared sulphate is not satisfactory,

**Roasting and Reaction.** A mixture of sulphate and oxide, together with unchanged sulphide, can be obtained by roasting the sulphide. It is, however, difficult to hit the right proportions, but the reactions may be brought about more or less completely and metallic lead obtained.

*Exp. 71.* Put 20 grams of rich galena in a roasting dish, and place the dish in the front part of a moderately hot muffle ; keep the powder well stirred with an iron stirrer. Note that the powder swells and roasts rapidly, but with a little care it can be prevented from clotting. Continue the roasting for 20 minutes. Take out the dish and transfer its contents to the crucible used for the last experiment. Add 5 grams of the raw ore, and proceed exactly as described above. When the bubbling has very nearly ceased pour into a round mould. Break away the slag and weigh the lead.

*Exp. 72.* Repeat the experiment as far as the conclusion of the roasting, then put the dish back into the hottest part of the muffle. The partly roasted mass will melt, and the reaction take place with separation of lead. When quite fluid, and when the metal can be seen in the centre, pour the contents of the dish into a round mould. When quite set, detach the slag, and weigh the metal button.

This experiment is an imitation of the Flintshire method of smelting rich galena. The two operations of roasting and reaction are conducted in the same furnace.

*Exp. 73.* Collect the slags from the three experiments, and melt them together in the crucible used for the reactions. Insert a strip of hoop iron, and leave for a few minutes in a hot fire. Well stir the liquid with the strip gripped in the tongs, and tap it on the side of the crucible to shake off any globules of lead which may adhere to it. Pour quickly into a round mould, as the iron sulphide solidifies rapidly. Detach the slag and weigh the button.

*Exp. 74.* Put a scorifier into a hot muffle, and drop into it the buttons of lead obtained in the four experiments. When

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the scoria nearly covers the metal, pour the contents of the dish into a round mould. When the lead has set, detach the slag, and hammer the metal, which is now soft and malleable.

**Zinc Blende** is the common ore of zinc and contains the sulphide,  $ZnS$ . The metal is obtained from it by roasting and reduction.

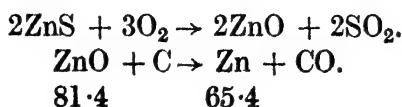
*Exp. 75.* Roast 5 grains of finely powdered blende. There is no danger of its clotting, and it only requires stirring occasionally. Continue the roasting until the odour of sulphur dioxide is no longer noticed.

The dark colour of the raw blende changes to reddish brown, which is due to the presence of iron sulphide in the ore.

*Exp. 76.* Weigh the roasted blende and mix with it one-third of its weight of powdered coke. Put the mixture into a thinly brasqued crucible, lute on a lid, leaving a small space free from clay. Put the crucible well down in a good fire, but so that the uncovered part of the lid can be seen. Close the furnace for 5 minutes, then draw the cover aside and watch the lid. A blue flame will be seen. This is due to the burning of the zinc vapour as it issues from the crucible. The zinc oxide formed in the roasting is reduced by the carbon at the high temperature of the furnace. When the change is complete, remove the crucible, and transfer the residue at the bottom, and any brasque adhering to it, to a roasting dish. Burn off the carbon in the muffle, and weigh the residue on the dish.

If the weight of the residue is subtracted from the weight of the roasted blende, the difference is the weight of zinc oxide in it.

The principal changes taking place in the operations are as follows :



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If then the weight of the oxide is multiplied by  $\frac{65.4}{81.4}$ , the weight of the metal in 5 grams of the ore is determined, and from this the percentage of the metal in the ore can be calculated.

## CHAPTER VI

### MECHANICAL TESTING

THE physical and mechanical properties of the metals and their alloys are very diverse, and require careful study, if the best use is to be made of them. The more important of these properties are colour, structure, hardness, density, fusibility, malleability, ductility, plasticity, toughness, elasticity, and tenacity.

**Colour.** The colours of the common metals and their alloys will have been already noted. With the exception of copper and gold the metals are white or white with a tinge of colour, and it is by this that a metal can be recognized and distinguished from one of similar appearance; as, for example, the faint yellow tinge of tin against the pure white of silver. With regard to colour changes, the effect of 5 per cent. aluminium on the colour of copper is very marked, as is also the whitening effect of nickel on the copper-zinc alloys. Also the effect of copper and silver respectively on the colour of gold may be cited.

**Structure.** Some information is to be obtained from the appearance of the fractured surface of a metal or alloy, but much more is forthcoming from an examination of the polished and etched surface under the microscope. The metal world owes much to metallo-

graphy, and its practical study should not be neglected by anyone interested in metals.

**Hardness.** The relative hardness of metals has assumed considerable importance of late, and various forms of apparatus for testing it are to be found in large works. The best of these is the Brinell hardness-testing machine, in which a hard steel ball is forced into the surface of the metal under known pressure. The measure of the hardness is obtained from the diameter of the circular edge of the depression caused by the ball. This is compression hardness, *i.e.* resistance to a blunt tool. For abrasion hardness a sharp tool under a known pressure is made to cut the metal away. The depth of the cut is the measure of the hardness.

**Density.** The determination of the density, or better of the ratio known as the specific gravity of metals, is often of importance. Thus if the specific gravity of the material is known, the soundness or otherwise of a small casting may be determined by taking its specific gravity.

The specific gravity of a solid is usually expressed as the ratio of the weight of the solid to the weight of an equal volume of water. The principle of the process lies in the fact that if a solid is immersed in water it displaces a volume of water equal to its own volume, and is supported in the water with a force equal to the downward pressure of its own volume of water. Therefore it must lose weight to the extent of the weight of an equal volume of water, and so weigh less than in air.

**Exp. 77.** Suspend a short piece of lead rod by a piece of thin cotton or silk from the hook above the scale pan of the balance. Support an empty beaker on a stool so placed that the scale pan can move freely, and arrange it so that the lead swings clear

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of the sides. Add weights to the weight pan until a balance is obtained. This gives the weight of the lead in air. Now pour water into the beaker until the lead is well covered. Examine it carefully for air-bubbles sticking to the surface. A simple plan is to brush the surface with a camel-hair brush after it is wetted, and so to ensure the absence of bubbles. Weigh again, when the lead will be found to have lost weight. Read the new weight, and calculate the specific gravity of the lead.

### EXAMPLE

$$\frac{\text{Weight of lead in air}}{\text{Loss of weight in water}} = \frac{75.32}{6.624} = 11.37 = \text{specific gravity of lead.}$$

*Exp. 78.* Determine the specific gravities of the aluminium-zinc alloys made in Exp. 121.

The specific gravities of alloys cannot be calculated from their percentage compositions and the specific gravities of their constituent metals. This could only follow from the metals occupying exactly the same volumes in the alloy as in the free state, and this is an unusual occurrence, for there is usually either contraction or expansion in total bulk on alloying.

**Malleability.** This is the property of extension in all directions without fracture between rolls or under the hammer. Hence there is evidently an extensive movement of the particles of the metal without break of continuity between them. It is a kind of flow of the solid, which must therefore assume a semifluid or viscous condition under pressure. But as a result of the work put into the metal it hardens and becomes more or less brittle, so that if the manipulation is continued beyond a certain point it cracks. The strain put into the metal by working is, however, relieved by annealing at the proper temperature, and it returns to its normal condition ready to receive more work.

The annealing process should be conducted at the

proper temperature, which varies for different materials, and oxidation of the surface of the metal should be prevented as far as possible. Heating to too high a temperature, or for too long a time, causes excessive crystallization or "burning" as it is called, and the metal becomes rotten. The higher the temperature at which the annealing is carried on, the shorter should be the time of exposure.

**The Rolls.** Hand rolls are very useful for experimental purposes, and the student should use them freely in testing the alloys he makes. For this purpose the castings are made in a strip mould. The ingots obtained from an open mould, being rough and uneven, are likely to damage the rolls as well as give an indefinite result. There are two rolls, the lower of which is driven and moves round in fixed bearings, while the upper one is carried by movable bearings, which can be raised or lowered by gearing connected with them. The space between the rolls is called the "pass," and the extent to which the upper roll is screwed down the "pinch." Too great a pinch should be avoided in any case, as the metal acts better if the work is put in slowly. As soon as the strip hardens, and before it shows signs of cracking, it should be annealed. The rolling may then be resumed.

*Exp. 79.* Melt 40 grams of lead under a little charcoal and pour it into the strip mould. Melt 38.5 grams of lead as before, and when the metal is at a moderate red heat, drop in 1.5 grams of antimony. Well stir the contents of the crucible with a charred stick, and, when the metal has cooled somewhat, pour it into the strip mould. Shear the strips to the same size, and trim the edges with a file. Put them through the same pass of the rolls, and gradually increase the pinch until the strip containing the antimony begins to crack on the edge. Place the

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two pieces across the hands, and press them in the middle with the thumbs. Make a note of the results of the experiments.

Two students should work the rolls together, one to turn and the other to pass. The difference in the amount of work put into the strips is easily detected by the one who is turning the rolls. The influence of the small amount of antimony in the lead is very marked. Lead and tin do not harden to an appreciable extent when rolled, but gold and silver do, and require annealing if much work is put into them.

**Ductility.** This is the property which allows a bar of metal to be extended in the direction of its length by a longitudinal stress, assisted by a side pressure at right angles to the direction of pull. For a metal to be very ductile it must possess a fair amount of cohesion between its particles in addition to its plasticity. That is, the metal must be tenacious as well as plastic. All that is required for testing the ductility of a metal is a draw-plate, which is a hard steel plate pierced by a series of carefully graduated holes, and a pair of pliers. The plate is fixed in the vice, and the metal is pulled bodily through the holes one after the other. It is much easier with a draw-bench, but two students are required for the work.

*Exp. 80.* Cut a narrow strip in the direction of the length of the cast strip to be tested. It should be a little wider than the thickness of the strip. Hammer it straight, and file it to a uniform, even section. Now file one end so that it will pass, through the largest hole in the draw-plate, and project sufficiently on the other side to be gripped by the draw-pliers. Pull it through the hole, and repeat the process until the strip is drawn into a wire about 12 in. long. It will probably require to be annealed two or three times during the drawing. Cut the wire into two, and anneal one piece.

**Tenacity.** The tensile strength of metals and alloys is measured by the resistance which a bar or wire offers to its fracture by a stress acting in the direction of its length. It requires then to be able to measure the stress applied and the cross-section of the bar under examination. An excellent machine for testing wires and thin rods is constructed by Messrs. Avery. The

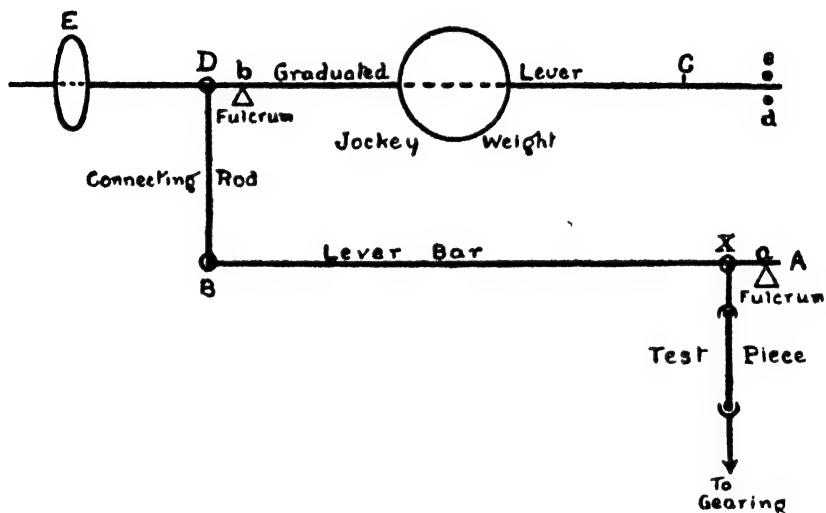


FIG. 22

principle upon which it works is shown in Fig. 22. It is a double-lever machine. The lower lever AB is supported on the fulcrum a, the upper one on the fulcrum b, and the two are connected by the rod c. When the jockey weight is in the zero position, CD is just balanced on b by the compensating weight E, so that it moves freely between the stops e d. If a stress is applied at X, B moves down, and this motion is communicated to D through the connecting-rod, and therefore C moves up between the stops. If now the jockey weight is moved towards C a position will

be found at which the arrangement is in equilibrium. That is, the stress is balanced. In this way any increase in the stress within the limits of the machine can be balanced by the jockey weight. As the upper lever is graduated, the measure of the stress in pounds can be read off at once from the position of the jockey. The upper grip for holding the test-piece is suspended from X, and the lower one is linked to a rod which works in a groove, and which can be raised or lowered by hand gearing below. If the grips are joined by a test-piece, and the gearing is worked so as to depress the rod, a stress is put upon the test-bar, which is measured by the balancing position of the jockey.

It is well known that the mechanical advantage of a lever is the ratio of the long arm to the short one. If, for example,  $aX$  is one-tenth of  $aB$ , a downward stress of 10 lb. at X is balanced by an upward pull of 1 lb. at B. Similarly if  $bD$  is one-tenth of  $bC$  the pull of 1 lb. at D is balanced by  $\frac{1}{10}$  lb. at C. From this it is clear that the advantage of the double lever is 100. Therefore a jockey weighing 50 lb. would balance and measure a stress of 5000 lb. on the test-bar. By hanging properly adjusted weights on the end of the upper lever this can be increased. With a larger machine worked by a hydraulic cylinder the stresses may be applied and measured in tons. This is the practice in the case of thick bars.

Four quantities can be measured and calculated. They are the *yield point*, or stress at which the test-piece begins to permanently elongate; the *breaking stress*; the *percentage elongation*; and the *reduction of area*.

Wires for testing are always circular in section; bars

are usually circular, but sometimes square or even rectangular in section. In all cases the area of the cross-section must be calculated. The measurements are made with a micrometer gauge. For a square bar two sides at right angles are measured, and if they differ by a small amount the mean of the two measurements is taken, and this, multiplied by itself, gives the cross-section. For a rectangular bar two sides at right angles are measured, and the two values multiplied together give the cross-section. For a wire or circular bar the diameter is measured, and if this measure is denoted by  $d$ , then :

$$\text{Area of cross-section} = d \times d \times .786.$$

The decimal .786 is a sufficiently close approximation to the ratio of the area of a circle to that of the square in which it can be inscribed.

The ends of the test-bars which are held by the grips are usually thicker than the middle part, which is worked to gauge. Two small centre-punch marks are made on the bar, and the section between these marks should be uniform. The distance varies from 2 in. according to the thickness of the bar. This is measured by calipers, and is taken as the length from which the percentage elongation is calculated. The bar is arranged in the grips, and the calipers are held in position in the two punch-marks, one point being held in firmly and the other loosely. The stressing is then commenced and the balance kept. When the yield point is reached, the punch mark will be drawn away from the loosely held point of the calipers. The value of the stress is noted. The stressing is then continued through the maximum load to fracture.

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The value of the breaking stress is noted. The two parts of the bar are taken from the machine, placed together, and the distance between the punch marks measured. The diameter of the bar at the point of fracture is also measured.

The tensile strength = 
$$\frac{\text{breaking load}}{\text{area of cross-section}}$$
 and is given in tons or lbs.

The elongation % = 
$$\frac{e \times 100}{l \text{ between marks}}$$
 when  $e$  is the increase in length between the marks.

The reduction of area is found by subtracting the cross-section found from the second diameter from that found from the first, or :

$$\text{Reduction of area} = (d^2 - d_1^2) \cdot 786 \text{ sq. in.}$$

As it is somewhat difficult to determine the yield-point and the reduction of area in the case of thin wires, these quantities will be neglected.

*Exp. 81.* Test the two wires prepared in Exp. 80, and set out the results according to the following scheme. Measure the length over all, and the diameter of the wires ; also the length between the grips when the wire is ready to be stressed. Measure the length over all after fracture.

Wire	Length over all	Length between grips	Diameter	Elongation	Elongation per cent.	Breaking stress	Breaking stress per sq. in.
Hard	6"	3 $\frac{1}{2}$ "	.0504"	1"	3.45	164"	37
Soft	6"	3 $\frac{1}{2}$ "	.0504"	1 $\frac{1}{2}$ "	20.7	141"	31.8

Similar tests may be made with thin rods having breaking stresses within the limits of the machine, the same measurements being recorded.

Tests of brass, mild steel, and cast-iron bars should be made the subjects of demonstration, if possible, when students are recommended to set out the results according to the following scheme :

Distance between marks	Diameter of bar	Diameter at fracture	Elongation	Elongation per cent.	Yield-point	Breaking stress	Tensile strength in tons	Reduction of area
2 "	.564"	.401	.74"	37	3.15	7.43	29.72	.126□"

The elongation of the test-bar also gives a good indication of the toughness and plasticity of the metal. These properties are very marked in good brasses, and are practically absent in grey cast-iron.

**Elasticity.** A metal under stress resists the deformation which takes place, and, up to a certain point, is able to regain its original form, when the deforming force is removed. This property is known as elasticity, and the point at which the original form of the body fails to be restored after removal of the force is called the *elastic limit*, beyond which permanent deformation sets in, as for example the permanent elongation of a test-bar. Experiments with metals in this direction require delicate apparatus, but the general property may be demonstrated in a simple way by means of a rubber tube arranged as shown in Fig. 23.

Two metal rods A, B, with hooks at one end, are secured to the ends of a thick rubber tube about 12 in. long. A strip of sheet metal C is soldered to the lower rod at right angles to it, and near to its junction with the rubber. A scale pan D is suspended from the lower hook, and the whole is hung by the upper

hook in a slot running the whole length of a strip of wood, which acts as a frame. A strip of plain paper is pinned to the side of the slot so that the metal strip can move over it when the rubber is stretched. The scale pan is arranged to carry a number of pieces of sheet lead weighing 6 grams each. These are readily made from sheet lead, and it is not important that they should be exactly 6 grams, provided that they weigh exactly the same.

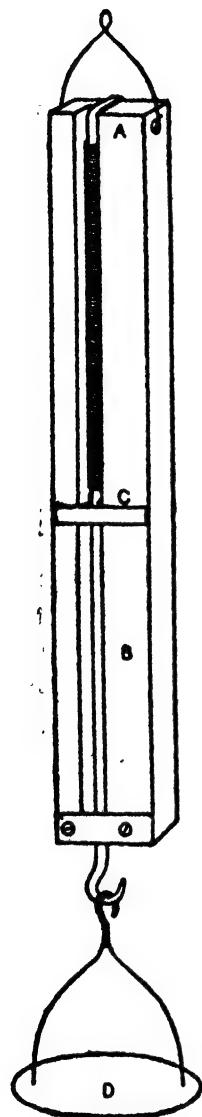


FIG. 23

*Exp. 82.* Hang the apparatus on a nail, or otherwise fix it in a vertical position. The weight of the scale pan will stretch the rubber sufficiently. Press the index against the paper with the thumb, and, using a pencil with a fine point, draw the initial line to show the position of the index. Place a weight on the scale pan, and draw a second line to mark the position of the index for the first stress. Continue until 10 to 15 weights have been added one by one. Remove the weights one by one, and note that the index does not quite return to its proper mark in each case. When they have all been taken off, the index shows that there is still strain in the rubber; but this is only temporary, and gradually dies out. Take off the paper strip, and draw a stress-strain diagram by means of it.

Take a sheet of plain paper, and draw two straight lines parallel with, and near to, two of its intersecting edges. The point where these lines meet is the origin. Beginning at the origin, mark off a number of equal spaces to represent the weights used. These correspond to equal stresses. Place the initial line on the paper strip on the origin, and the edge along the

horizontal line. Mark spaces along it corresponding to the spaces on the strip. These represent strains in the rubber. Find the points of intersection of vertical and horizontal lines passing through corresponding stress-strain points on the reference lines. This is readily done with a piece of stiff paper, if its horizontal edge is made to cut through the first stress point, and its vertical edge through the first strain point. A dot made with a pencil at the point where the two edges intersect marks it. Do this for each pair of points on the reference lines,

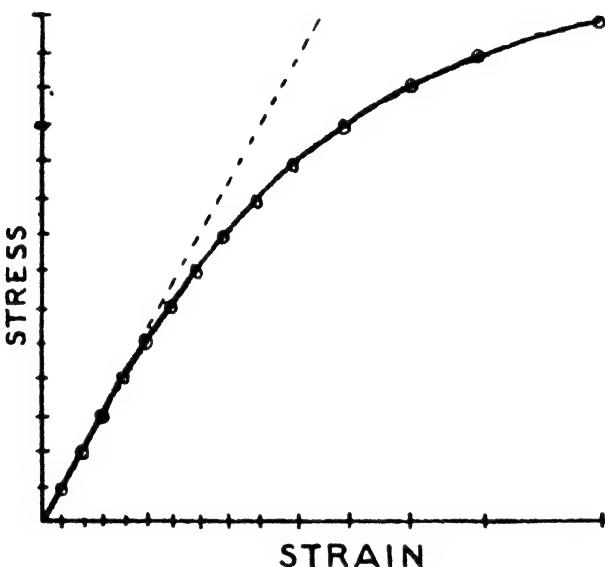


FIG. 24

and connect the dots by a continuous line drawn from the origin through them. This is the stress-strain curve for the rubber tube.

It will be noticed that the lower part of the curve is practically a straight line. This is shown clearly by placing a rule with its edge passing through the origin and the dots as far as they lie upon it, and then drawing a line through them as shown in Fig. 24. The upper part of the curve is seen to fall away as soon as the proportionality between the stress and strain varies.

The analogy between steel and rubber is not by any means perfect, but a general idea of the nature of the curve is obtained in a simple way. The point where the curve begins to fall away is analogous to the yield-point in the case of a steel rod. The elastic limit is also in this region.

The *fatigue* which is so noticeable in the rubber tube is also present in steel and other metals when they are stressed, but is only in evidence within the elastic limit. It may be defined as the inability of the body to recover its form at once, when the deforming force is removed.

**Transverse Strength.** Tests of the strength of bars across the direction of their length is only applied to cast-iron, but is regarded in that case as more important than the tensile test. The testing machine is based on the lever principle, which is shown in Fig. 25. The long arm of the lever AB, which is supported on the fulcrum C, is graduated, and carries the jockey weight. It is balanced, when the jockey is at zero, by the dead weight E. The screw F, after passing through fixed and loose collars, presses on the knife edge G, which limits the length of the short arm of the lever. A stirrup descends from the loose collar and carries a link by which the test-bar is drawn up against the rigid knife edges in *dogs*, or eye-pieces, H, J. When the screw is turned so that the loose collar moves upwards, it pulls the link against the under side of the test-bar, and stresses it across the direction of its length; and if AB is kept horizontal by moving the jockey towards B, the stress is measured. The hand-wheel at the top of the screw is turned slowly, and the jockey is moved until the bar breaks. The position

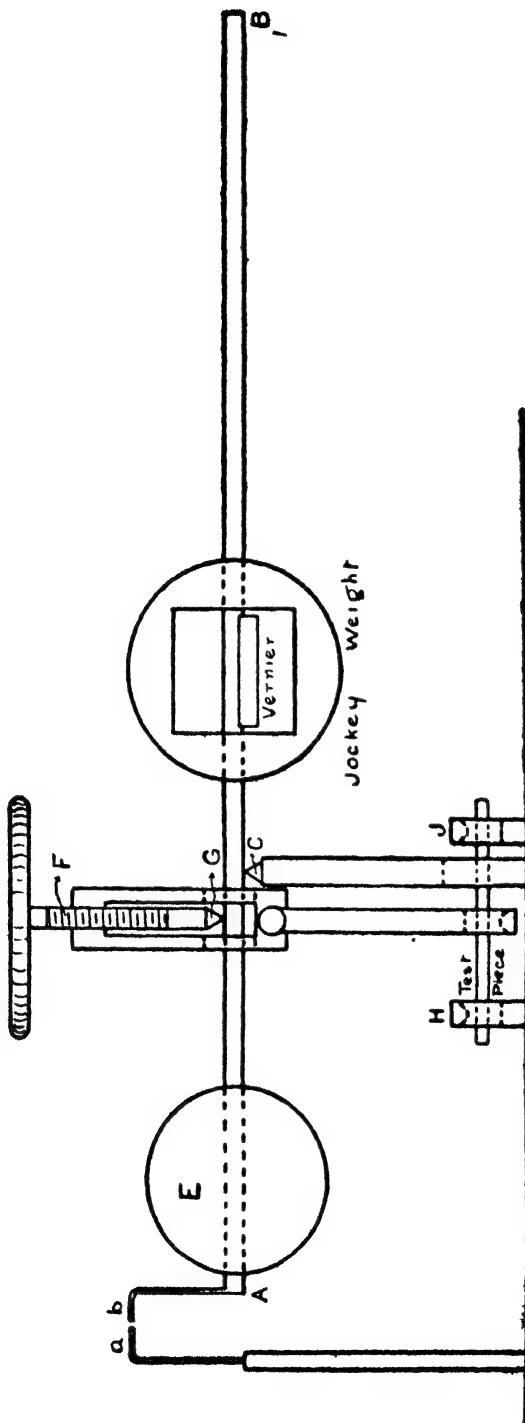


FIG. 25

of the jockey indicates the breaking stress in cwts. A vernier is attached for reading fractions of a cwt., and the loose collar carries a scale for measuring the deflection of the bar at the moment of breaking. The lever is horizontal when the two points *a b* are in line. Hence they must be watched continuously while the test is proceeding.

The test-bars usually specified are 3 ft. 6 in. long, 2 in. wide, and 1 in. thick. They are placed in the machine with the fixed knife edges 3 ft. apart, with the loose knife edge just under the middle point, and the long side vertical. Such bars should break at a stress of from 25 to 28 cwt., but very good metal will sometimes give a breaking stress of 30 cwt. These bars, however, can only be cast satisfactorily in a foundry, so that smaller ones are more suited for experimental work. A bar 14 in. long and 1 in. square may be used and can be readily cast in the laboratory. Such bars should break with stresses varying from 18.7 to 22.5 cwt. The reason why the 1 in. bar gives a relatively higher breaking stress than the longer and wider bar is to be attributed to the shorter length under stress. Square bars 14 in. long and .75 in. on the side have been successfully used for determining the influence of silicon and other elements upon the transverse strength of cast-iron. About 3 lb. of metal are sufficient for a good cast.

The moulding and casting of bars for transverse and tensile tests can be readily carried out in a laboratory furnished with the necessary apparatus. This consists of a rectangular moulding tub nearly filled with casting sand, and furnished with a strong cover in two parts, on one of which the mould is made; a moulding flask,

which consists of rectangular iron frames about 2 in. deep, open at top and bottom, and furnished one with pegs and the other with eyes, so that they can be readily put together or taken apart; flat boards, rather larger than the flask, with a wooden frame and screw for clamping the parts together; and a number of trowels and rammers of various shapes. The actual process of moulding is to be carried out under supervision, when bars of cast-iron and of non-ferrous alloys may be prepared for testing.

## CHAPTER VII

### NON-FERROUS METALS AND ALLOYS

A PRACTICAL knowledge of the physical and mechanical properties of the common metals and their alloys, and of the preparation of alloys, is of much importance in dealing with the many changes they pass through in various manufacturing processes. The use of pure metals alone, although of some importance, is limited compared with that of their various alloys. So that more attention will be given in this chapter to the alloys than to their constituent metals.

An alloy may be defined as a complex substance having the physical and mechanical properties of a metal. It may consist of an intimate mixture of the constituent metals, of a solid solution of one metal in another, or of a chemical compound of two metals. Or there may be two or more of these definite associations of the metals in the same alloy. Also non-metals such as carbon, silicon, and phosphorus are present as essential elements in the composition of some alloys.

The internal structure of alloys upon which their useful properties depend, and the effects of various kinds of treatment they have to pass through, have received much attention during recent years, and with useful results. The constituents of the structure of an alloy are to be distinguished from the constituent

metals themselves. The more important and well defined of these structural constituents may be briefly described.

The *single solid solution* consists of a solution of one metal in another, or of a compound of the two metals in one of them. It has the same structure as that of a pure metal, and its working properties are those of its constituents in a modified form. Alloys which are made up entirely, or for the most part, of single solid solutions have excellent working properties. This is shown clearly in the case of the brasses containing more than 66 per cent. copper.

The *eutectic* alloy is an intimate mixture of the two metals in definite proportions which is deposited at a definite temperature on the final solidification of the cooling mass. An alloy having the exact eutectic composition has a sharply defined melting-point which is lower than that of any other alloy of the same metals containing an excess of either metal over the eutectic composition. It is the easily melted alloy of the series. Eutectics are not present in all alloys, but are formed sometimes from a solid solution and excess of one of the metals, or from a chemical compound of the two metals and excess of either. Tin-lead and copper-antimony eutectics are notable examples. The eutectic is a well-defined structural constituent of alloys.

The *chemical compound* in its pure state is brittle and unworkable, and is therefore of no manufacturing importance. When present in small quantities in alloys it modifies their properties, sometimes usefully, as is the case with bearing metals.

**Melting.** Strict attention to the furnace is necessary for good work in melting metals. The pieces of coke

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should not be large compared with the size of the crucible, nor so small as to interfere with the draught. The fire should be kept in good condition to prevent waste of time. Do not leave the fire to burn low while preparing an experiment. Make it up, if necessary, before leaving it.

*Exp. 83.* Weigh out 50 grams of lead, and melt it in an open clay crucible in a moderate fire. Take out the pot and allow it to stand on the furnace top till the redness has quite disappeared. Pour the metal carefully into a short rectangular mould. Thoroughly rub the inside of the pot with a charred stick, and tip the débris on to the plate. Weigh the ingot and note the loss during melting. Make it up to 50 grams again with fresh lead. See that the crucible is clean from the last melt; put in the lead with a little charcoal, just sufficient to cover the metal when melted; put a lid on the pot, and proceed exactly as before. Weigh the ingot and compare with first ingot.

In melting lead in an open crucible the surface of the metal is exposed to the air and oxidation takes place with consequent loss of metal. When the metal is covered with a layer of charcoal this cannot take place and loss is avoided. If the metal is poured at too high a temperature the surface of the ingot shows signs of oxidation, and a bad cast is the result.

*Exp. 84.* Melt 40 grams of copper in a clay crucible with the lid on. The fire must be good, and the pot placed well down in it, or the metal will take too long to melt. Close the furnace and leave for 10 minutes. Take off the lid and pull the pot up a little in the fire. If the copper is melted, note its bright metallic appearance when the lid is taken off, and then the rapid dulling of the surface. Shake the crucible, when the dullness will disappear. Repeat twice; put on the lid and close the furnace for a minute; pour the metal rapidly into a short rectangular mould. Weigh the ingot and break in vice.

The surface of the molten copper absorbs oxygen rapidly when exposed to the air with formation of red oxide of copper,  $Cu_2O$ , which dissolves into the molten metal. It is said to be "dry," and if sufficient oxygen has been absorbed shrinks in the mould. A furrow is thus formed along the middle of the ingot. The fractured surface is dull red and dry looking.

*Exp. 85.* Melt 40 grams of copper under sufficient charcoal to cover it when molten. Keep molten for 10 minutes, then pour into the rectangular mould, but keep the charcoal from the surface of the ingot. This can be done by pushing it aside with a thin iron poker. Weigh and fracture the ingot.

The charcoal, if in contact long enough, removes the small quantity of oxygen required to keep the metal tough, with the result that it breaks short, and the fracture shows large, bright grains. It can be toughened again by putting in a little oxygen, but it is by no means easy to introduce just the right quantity by exposing the molten metal to the air for a short time.

*Exp. 86.* Melt 30 grams of zinc under charcoal at the lowest possible temperature. When quite molten pour it into a rectangular mould. Weigh the ingot, remelt it under charcoal, and raise to a bright red heat. Note the blue flame inside the crucible, and the white fluffy deposit on the sides. Allow to cool a little, pour, and weigh the ingot.

*Exp. 87.* Repeat the experiment with 20 grams of tin, and note the great difference in behaviour of the two metals.

Zinc melts at  $420^{\circ} C.$  and begins to vaporize at a red heat; it boils at  $940^{\circ} C.$  with rapid *liberation* of vapour. When the vapour comes into contact with the oxygen of the air it burns and forms zinc oxide,  $ZnO$ . Tin melts at  $232^{\circ} C.$ , but requires a much higher temperature than zinc to vaporize it, and even then the change from liquid to gas is slow. Consequently there

is very little loss when the metal is heated to a bright red heat out of contact with air.

**Alloys.** The general method of preparing alloys is by melting the constituent metals together to form a homogeneous molten mass, and casting the same at the proper temperature, and under the best conditions to ensure sound ingots or moulded castings. There are various causes of faulty casts. The casting temperature may be too high or too low ; gases absorbed by the molten metal may be liberated at the moment of solidification and cause blow-holes ; gases from the air and dirt from the crucible may be carried into the mould during pouring, and so cause defects.

For success in making alloys on the small scale it is necessary to observe some general rules which may be stated now and which should be carefully noted. (1) The furnace should be attended to and the fire made up before weighing out the metals, so that it will be ready for use as soon as the experiment is prepared. (2) When two metals having widely different melting-points are to be alloyed, the metal having the higher melting-point should be melted first, and the other added when the first is well above its melting-point so as to prevent too much chilling by the addition of the cold metal. The larger the proportion of the second metal the greater the care required in its addition ; and it may be useful to hold it in the top of the pot to get warm before allowing it to drop in. Sometimes the addition of the cold metal causes the hot one to solidify, in which case the remelting and alloying are never quite so satisfactory. (3) The surface of the metal must be just covered with charcoal either finely or coarsely powdered. It is better to add too little than too much

at first, as more may be added if necessary. It is difficult to pour the metal when much charcoal is in the pot. Charcoal should be added in most cases, except with alloys rich in aluminium.

**Copper-tin Alloys.** There are very few alloys consisting of copper and tin alone. The best known of these are bronze and bell metal. Tin and copper form a compound  $\text{Sn.Cu}_3$ , which dissolves in excess of copper forming a single solid solution, when the tin does not exceed 10 per cent. This alloy has excellent working properties. The finest alloy contains about 9 per cent. tin. Bell metal is brittle and can only be cast and dressed, but its sound properties are good. If quenched at a low red heat it becomes lighter in colour and somewhat malleable.

*Exp. 88.* Melt 36.4 grams of copper under a little charcoal, and add 3.6 grams of tin. Shake the crucible to ensure thorough mixing ; close the furnace for a minute ; pour the melt quickly into a rectangular mould as the alloy chills rapidly. Weigh the ingot, and break it in the vice. Calculate the composition of the alloy from the weights of metals taken. Reserve the ingot for comparison with phosphor bronze, Exp. 103.

*Exp. 89.* Weigh out 32 grams of copper and 8 grams of tin and proceed exactly as above. Break the ingot across the middle ; raise one-half to a moderate red heat and quench it in water. Test the two pieces on the anvil. The quenched piece should flatten more easily than the other.

Bell metal varies considerably in composition according to the use to which it is to be put. The range may be said to be 75-84 per cent. copper and the remainder tin.

**Copper-zinc Alloys.** This series is the most important among the copper alloys. Brass, on account of its excellent working properties, is used for a variety of

purposes. The formation of solid solutions is the most prominent feature of the copper-zinc series, and all the alloys with more than 66 per cent. copper consist of a single solid solution having the property of being workable in the cold. Those containing about 60 per cent. copper can be rolled better at a red heat, below which the solid solution breaks up and the alloys lose their malleability to some extent. Alloys containing 50 per cent. and less are more or less brittle and cannot be worked. There is no advantage in increasing the copper content beyond 70 per cent. as far as working properties are concerned ; but the colour is a little deeper, and alloys containing upwards of 80 per cent. copper are made for such purposes as cheap jewellery and ornamental brass work.

*Exp. 90.* Gilding metal, 82.5 per cent. Cu and 17.5 per cent. Zn. Weigh out 33 grams of copper and 7.5 grams of zinc. Have the zinc in one or two pieces only, if possible, as it is much easier to introduce than in a number of small pieces. Melt the copper in a clay crucible under a little charcoal. Put the pot well down in the fire with the lid on, so as to get a rapid melt. When the copper is melted, raise the pot a little in the fire, and place the lid on the coke beside it. Grip the zinc in the tongs and hold it in the top of the pot for a few seconds to warm, then drop it into the copper and quickly replace the lid. Push the lid aside just sufficiently to insert the tongs, and shake the pot to mix the metals. By keeping the lid on as much as possible loss of metal by spurting on account of the rapid escape of zinc vapour is prevented. This is often due to the fact that too much charcoal on the surface of the molten metal prevents the zinc from melting into the copper at once. Close the furnace for a few seconds, and then pour the melt into the rectangular mould. Clean the pot with a charred stick, and collect any shots of alloy left. Weigh them with the ingot. Break the ingot and examine the fracture.

With care there is no difficulty in making small

ingots of alloys containing zinc. The principal cause of loss is the volatilization of zinc, which must be allowed for if the alloys are to be of definite composition. It will be noticed that .5 gram of zinc is allowed for loss in making the above alloy. This amounts to about 1 per cent. on the full weight. As a general rule 1 per cent. may be added for every 15 per cent. of zinc for small ingots. With larger weights of metal the percentage loss of zinc is not so great. Charcoal must be used sparingly, as it is the common cause of trouble in adding the zinc.

If no spouting takes place in preparation, the exact composition of the alloy can easily be found from the weight of the ingot, as the whole of the copper put into the pot is in the ingot and shots.

$$\frac{\text{Weight of copper}}{\text{Weight of alloy}} \times 100 = \text{percentage of copper in alloy.}$$

Proceed exactly as above in making the following alloys :

*Exp. 91.* Cartridge metal, 70 per cent. Cu, 30 per cent. Zn. Weigh 28 grams of copper and 12.8 grams of zinc.

*Exp. 92.* Yellow brass, 66 per cent. Cu, 34 per cent. Zn. Weigh 26.4 grams of copper and 14.5 grams of zinc.

*Exp. 93.* Muntz metal or yellow metal, 60 per cent. Cu, 40 per cent. Zn. Weigh 24 grams of copper and 17.1 grams of zinc. Cast in a strip mould.

*Exp. 94.* Brazing solder, 50 per cent. Cu, 50 per cent. Zn. Weigh 25 grams of copper and 26.5 grams of zinc.

These alloys are important members of the copper-zinc series on account of their excellent working properties. With the exception of brazing solder, they are malleable and ductile, but Muntz metal rolls better at a red heat; it can also be rolled cold. It is well

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known that the structure of copper-zinc alloys passes through a temperature range somewhat below a red heat. The metal becomes comparatively rotten, and is readily broken up. This result is more marked as the content of zinc increases, and brazing solder furnishes a good example. The granular powder used for hard soldering is made by taking advantage of this property.

*Exp. 95.* Heat half the ingot of brazing solder made in the last experiment to a red heat; drop it into an iron mortar, and pound it with the pestle as it cools down. If the pounding is successful the metal will break up into fragments, but this depends upon the blows being delivered at the proper temperature.

While it is not possible to increase the malleability and ductility of copper-zinc alloys by the introduction of a third metal, their hardness and tensile strength may be considerably increased by such an addition. The important additions are iron, manganese, and sometimes aluminium. Thus the hardness and strength of the 60-40 alloy is considerably increased by the addition of a small percentage of iron. Delta metal, which was patented by Dick, is a typical alloy of this class. Dick's idea was to introduce the iron in the form of an alloy of iron and zinc, but this appears to be a wasteful method, in that there must be a considerable loss of zinc in making the iron alloy. An alloy of iron and copper is now largely used. Commercial delta metal often contains manganese and aluminium in addition to iron. Aich metal and sterro metal are similar alloys.

*Exp. 96.* Weigh out 23 grams of short pieces of rod copper, and .6 gram of thin iron wire which has been cleaned by rubbing with emery cloth. Binding wire is very suitable for the purpose.

Cut the wire into as many pieces as there are pieces of copper, and wind each tightly round a piece of copper, and make secure by twisting the free ends with pliers. Transfer the whole to a clay crucible with a little charcoal, put it well down in a good fire, and cover it with a lid. Make up the fire, close the furnace and leave it for from 20 to 30 minutes. Shake the crucible, and pour the alloy into an open mould. Examine the metal and the charcoal débris in the crucible to see if the metals are properly alloyed. Fracture the ingot and note the appearance of the fractured surface. Remelt the copper-iron alloy, and add 17 grams of zinc with the usual precautions. Pour the alloy into a rectangular mould; weigh, and break to examine the fracture.

*Exp. 97.* Weigh out 23 grams of copper and 1 gram of high-grade ferro-manganese in small pieces (not powder). Transfer to a clay crucible together with a little charcoal, and proceed exactly as in the last experiment.

The fractured surfaces of these alloys show a closer grain and a lighter colour than ordinary Muntz metal.

Brasses containing only copper and zinc are somewhat difficult to turn on account of their toughness, which causes the turnings to run to considerable length and to clog the tool. It is found, however, that the addition of 1-2 per cent. of lead makes the alloy much more workable under the tool. The lead does not form a solid solution, but separates in the free state throughout the mass, and so reduces the toughness that the turnings break off much shorter and leave the tool readily.

*Exp. 98.* Weigh out 26 grams of copper, 14.5 grams of zinc, and .9 gram of lead. Melt the copper under a little charcoal, add the lead, and then the zinc with the usual precautions. Pour into a rectangular mould; weigh and fracture the ingot.

On comparison of the fractured surface with that of the 66-34 alloy, the former is seen to have a slightly

duller appearance, due to the particles of lead distributed through it. Also if the two pieces are tested with a file, the one containing the lead drags less under it. Brass castings which do not require to undergo mechanical treatment, except turning, filing, and screwing, usually contain relatively large amounts of impurities such as lead, tin, and iron. This results from the use of common scrap in making the alloys.

**Copper-nickel Alloys.** These metals alloy together in all proportions to form solid solutions, which are rendered homogeneous by careful annealing. Thus, while the alloys have the structure and general properties of a single metal, they are harder and more tenacious than copper. But they find a limited use on account of the price of nickel. A well-known alloy, "Constantan," containing 60 per cent. copper and 40 per cent. nickel, has important electrical properties. As a rule these alloys do not contain more than 25 per cent. nickel, and are known under the name of cupronickel. A 50-50 alloy is used in the manufacture of nickel silver.

*Exp. 99.* Weigh out 35 grams of copper and 15 grams of nickel, which should be in small pieces. Transfer to a clay crucible with a little charcoal, place in a good fire, put on a lid, and cover with coke. Heat strongly for 30 minutes. Stir with a charred stick to see that the metals are properly alloyed, and pour into a strip mould. Weigh the strip, which should be nearly full weight.

The fractured surface of the alloy shows a close-grained structure and a greyish-white colour. The strip is easily cut up for making the next series of alloys, and the difficulty of alloying the copper and nickel is overcome once for all.

**Copper-nickel-zinc Alloys.** Different makes of these alloys are known by a variety of names, such as nickel silver, potosi silver, silveroid, etc. They have excellent working properties, but are harder and stronger than the brasses. The presence of the nickel reduces the brassy colour and, if in sufficient quantity, entirely removes it. The best qualities are white. If time permits, the following series should be made, using the cupro-nickel prepared in the last experiment to introduce the nickel. To do this, calculate the weight of nickel to be introduced, the weight of cupro-nickel to carry it in, the weight of extra copper, and the weight of zinc. This will be best illustrated by an example. The method may be used for calculating the crucible charge in which an alloy, either scrap or new metal, forms part of it :

To make a 40-gram ingot of nickel silver containing 16 per cent. nickel, 56 per cent. copper, and 28 per cent. zinc, using a cupro-nickel containing 30 per cent. nickel.

$$\begin{array}{lcl} \text{Cupro-nickel required} & = 16 \times \frac{2}{5} \times \frac{10}{3} & = 21.4 \text{ grams} \\ \text{Nickel present} & = 16 \times \frac{2}{5} & = 6.4 \text{ } , \end{array}$$

$$\begin{array}{lcl} \therefore \text{Copper } , & = & 15.0 \text{ } , \\ \text{, to make up } & = & 7.4 \text{ } , \end{array}$$

$$\begin{array}{lcl} \text{, in alloy} & = 56 \times \frac{2}{5} & = 22.4 \text{ } , \end{array}$$

$$\begin{array}{lcl} \text{Zinc required} & = 28 \times \frac{2}{5} + 1 & = 12.2 \text{ } , \end{array}$$

*Exp. 100.* Weigh 21.4 grams of 30 per cent. cupro-nickel and 7.4 grams of copper ; transfer to crucible with a little charcoal, and melt in a good fire with the lid on. Weigh 12.2 grams of zinc, and proceed exactly as in making a brass. Pour the alloy into an open ingot mould, and weigh the ingot and any scraps.

Make two similar ingots containing nickel 10 per cent., copper

56 per cent., zinc 34 per cent.; nickel 6 per cent., copper 58 per cent., zinc 36 per cent.

When the ingots are nicked with the chisel and broken, the fractured surfaces show clearly the influence of increasing the percentage of nickel. The best alloy contains 30 per cent. Ni, 50 per cent. Cu, and 20 per cent. Zn; it is a white metal having excellent wearing properties and taking a beautiful finish; but is somewhat difficult to work. These rich alloys are not used generally for electro-plate ware. The 10 per cent. alloy is excellent for that purpose, but better qualities are sometimes employed.

**Phosphor-bronze and similar Alloys.** The modification in the properties of an alloy by the introduction of a small quantity of a third metal has already been dealt with, and it now remains to consider the effects of the non-metal phosphorus in this respect. Phosphorus is a well-known deoxidizer, and as such has been in use for some time. In this case, however, the aim should be to introduce just sufficient phosphorus into the alloy to remove both the oxygen and itself at the same time. The phosphoric oxide formed by the reduction of oxide in the alloy combines with more oxide to form a phosphate, which, not being soluble in the molten metal, passes out in the form of a slag. It is thus seen that if the proper quantity of the non-metal is put in, the finished alloy will be quite free from it. This is the ideal case, and it frequently, happens that so-called phosphor-bronzes are quite free from phosphorus, but the result is the best possible for metal which has to be rolled. Excess phosphorus remains in the alloy as phosphide of one or both metals. With up to 2 per cent. phosphorus the alloy can be

cast into the required shape and finished by filing and turning, but cannot be rolled. Such alloys make excellent bearings for heavy machinery, but for this purpose often contain lead and zinc.

The phosphorus is most conveniently introduced in the form of either phosphor-copper or phosphor-tin. These alloys are produced commercially with guaranteed percentages of phosphorus; phosphor-copper containing up to 15 per cent. and phosphor-tin up to 10 per cent. of the non-metal. They are readily made on the small scale, but it is not necessary to aim at the richer alloys, as special methods are required to prevent excessive loss of phosphorus. There are two forms of phosphorus, the yellow crystalline and the red amorphous variety. The former is very inflammable and requires to be kept in water, but the latter may be freely handled and only takes fire when heated. This, though more expensive, should be used, unless under special conditions.

*Exp. 101. Phosphor-copper.* Llosely pack 25 grams of thin sheet copper in a clay crucible, and raise quickly to a red heat, with the lid on. Remove the lid and shoot in 2.5 grams of red phosphorus from a cold iron shoot; quickly replace the lid, and close the furnace. When the contents of the crucible are melted, pour into a rectangular mould. Weigh the ingot together with any shots. With care in making, the increase in weight of alloy over the weight of copper multiplied by 4 gives the percentage of phosphorus in the alloy approximately. The ingot breaks readily, and the colour of the fractured surface varies from grey to bluish-grey.

*Exp. 102. Phosphor-tin.* Put 2 grams of red phosphorus into a clean clay crucible, and tamp it down with the wooden end of the pestle. Melt 20 grams of tin in another crucible without charcoal, and when melted pour it on to the phosphorus. Put the crucible in the fire, and leave until the flame has disappeared; pour the alloy into an open mould. Weigh the ingot

and break it. The fracture shows brilliant crystals the size of which depends to some extent on the rate of cooling.

Two alloys illustrating general practice may be made from these materials : (1) In which but a small percentage of phosphorus is present. This alloy is malleable and has considerable tensile strength. It also resists corrosion very well, and on that account is used for the moving parts of machinery working in liquids. (2) Cast phosphor-bronze which may contain up to 2 per cent. phosphorus and 10 per cent. tin with lead in addition. This is a good bearing metal for heavy work. Zinc also often forms the third metal in phosphor-bronzes of both classes.

*Exp. 103.* Assume that the phosphor-tin made as above contains 5 per cent P. Weigh 2 grams of tin, 1·6 grams of phosphor-tin, and 36·4 grams of copper. Melt the copper with a little charcoal, and add the tin and phosphor-tin. Close the furnace for a minute. Shake the crucible well and pour the melt into a rectangular mould. Weigh and fracture the ingot. Compare for colour and closeness of grain with the result of *Exp. 88.*

*Exp. 104.* Assume that the phosphor-copper contains 5 per cent. P. Weigh 22 grams of copper, 10 grams of phosphor-copper, 4 grams of tin, and 4 grams of lead. Melt the copper and phosphor-copper together under charcoal, and add the tin and lead. Proceed as above. Calculate the percentage of phosphorus present, if  $\frac{9}{10}$  of that added remains in the alloy.

**Aluminium-bronze.** Alloys of copper containing up to 10 per cent. aluminium have very useful properties, but beyond that they are brittle and difficult to work. Those containing less than 7·5 per cent. consist of a single solid solution and can be readily rolled and drawn in the cold. With more than 7·5 per cent. a second and harder constituent appears in the structure which

increases the strength but decreases the malleability of the alloy. With more than 10 per cent. the alloys are of little use commercially.

*Exp. 105.* Melt 38 grams of copper with two small pieces of charcoal in the crucible and with the lid on. Add 2.1 grams of aluminium rod, and close the furnace for a minute or two. Shake the crucible well to obtain thorough mixing, and pour the alloy into a rectangular mould. Weigh the ingot and break it to examine the fracture. Remelt the broken ingot and cast in the strip mould. Reserve the strip.

Some metals, though well mixed when in the molten state, separate either partially or completely on solidification. This is due to the fact that one metal is soluble in the other in the solid state only to a limited extent, or not at all; also to the absence of eutectics in the structure of the alloys. Further, some metals form well-defined compounds with each other, and their alloys in these proportions are brittle and quite useless for ordinary purposes.

*Exp. 106.* Melt 40 grams of lead under a little charcoal, and when the crucible is at a moderate red heat add 20 grams of zinc. As soon as the zinc has melted, stir well with a charred stick, and pour the alloy into a rectangular mould. Grip the ingot in the vice with the top towards you, and carefully bend it over with the hammer. Note that the top part of the ingot breaks readily and shows the crystalline fracture of zinc, while the bottom is tough and bends over. File the side of the ingot. The bottom part is soft and clogs the file; the top is harder and brighter.

The lead having the greater density sinks to the bottom, and the zinc rises to the top. It is found, however, that the lead contains about 1.5 per cent. of zinc, and the zinc 1.5 per cent. lead in solid solution.

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*Exp. 107.* Melt 28 grams of copper with a little charcoal, and add 12 grams of lead. Stir the alloy thoroughly with a charred stick, and pour rapidly into a cold button mould. Repeat the experiment, but have a larger crucible and lid at a bright red heat ; put the crucible with the alloy in it into this, put on the lid and cover the whole with hot coke from the furnace. Remove the button when cold. Saw the buttons in two with a hack-saw, and compare the exposed surfaces.

Lead and copper do not form either a solid solution or a eutectic alloy, hence the lead tends to separate and sink slowly towards the bottom of the button. The longer the alloy is in passing from the liquid to the solid state the more complete is the separation of the two metals. Therefore the exposed surface of the alloy which was slowly cooled should show a greater concentration of lead towards the bottom of the button than that of the one quickly cooled. The separation is not as perfect as with lead and zinc.

*Exp. 108.* Melt 15 grams of copper with a little charcoal, and add .25 gram of bismuth. Well stir the alloy, and allow it to cool slowly as described above. Examine the button when cold.

Bismuth, when in excess, separates more completely from copper than does lead, and the surface of the button sometimes shows small globules of extruded bismuth. If one of these is removed and tapped with a hammer it breaks up into fragments of a crystalline metal with a reddish tinge. This is pure bismuth. That which remains more intimately associated with the copper separates round the copper crystals and forms a brittle cement which makes the alloy un-workable. This may be shown by melting the button, and casting it into a rod in a sand mould. The rod breaks readily in the vice. Bismuth is the most

injurious impurity in commercial copper, and the most difficult to eliminate.

*Exp. 109.* Melt 15 grams of copper with a little charcoal ; add 15 grams of antimony, and close the furnace for a minute or two ; stir the molten alloy, and pour it into a rectangular mould. Weigh the ingot ; hold it in the hand, and strike it with the hammer. It breaks readily, being very brittle, and the fractured surface shows a violet crystalline structure.

This is an excellent example of chemical combination between two metals. The compound is  $SbCu_2$  and contains 51.45 per cent. Cu and 48.55 per cent. Sb. A slight excess of antimony is used in the preparation as the metal is somewhat volatile. The alloy is quite useless even for ornamental purposes as it is so brittle that it cannot be dressed without portions of the surface breaking away.

The influence of a small quantity of a second metal on structure is well shown by the introduction of aluminium into zinc, and of arsenic into tin. In the first case the large crystalline fracture of zinc is considerably reduced, and in the second the buttery fracture of tin is rendered distinctly crystalline.

*Exp. 110.* Melt 40 grams of zinc at a moderate temperature, and pour it into a rectangular mould. Melt another 40 grams of the metal, and add 1 gram of aluminium to it. Stir well and pour into the same mould. Break the two ingots, and compare the fractured surfaces.

*Exp. 111.* Melt 30 grams of tin with a little charcoal, and pour at a low temperature into a short rectangular mould. Repeat, with 30 grams of tin, and add 3 gram of arsenic to the molten metal. Stir well and cast into a short ingot. Break the ingots, and compare the fractured surfaces.

**White Metals.** A number of useful alloys are comprised under this term. The best known of these are

## 100 ELEMENTARY PRACTICAL METALLURGY

the lead-tin solders. The lead-tin series is also interesting, as it is the one with which Guthrie worked in his original investigation of eutectic alloys. When two metals do not form either a solid solution or a compound, but simply mix together, there is generally one alloy which has a lower melting-point than either of the constituent metals or any other alloy of the series. With the lead-tin series this is the alloy containing 63 per cent. tin and 37 per cent. lead with a melting-point =  $180^{\circ}$  C., that is  $52^{\circ}$  lower than the melting-point of tin. It has this constant melting-point, and any other lead-tin alloy will contain it with excess of one or other of the metals. Now the peculiarity is that, on cooling any other alloy, the excess of either lead or tin will solidify and crystallize at a temperature above the eutectic point. So that the solid particles will be wetted by the still liquid eutectic, and the whole mass will be more or less pasty according to the proportion of the solid metal present, until the eutectic finally solidifies at  $180^{\circ}$ . This property is taken advantage of by the plumber in the ordinary process of "wiping" a joint in which the pasty material, an alloy rich in lead, is worked round the cleaned joint with the protected hands. The solder used by plumbers is approximately 2 parts lead to 1 part tin. It thus contains 46 parts of free lead and 54 parts of the eutectic composition in the pasty material. On the other hand, tinman's solder has nearly the eutectic composition and remains quite liquid down to its solidifying-point. This much facilitates its use for soldering purposes.

It should be noticed that a solid solution or a chemical compound in the vice. Two metals may form a

eutectic with excess of one or other or of both of the metals. It simply happens that in the case of lead-tin there is no chemical combination between the metals, and if there is a solid solution, it is but a weak one of tin in lead. Eutectics must be regarded as well-defined constituents in the structure of a number of series of alloys.

*Exp. 112.* Make two ingots, (1) containing 25.2 grams of tin and 14.8 grams of lead ; (2) 14.8 grams of tin and 25.2 grams of lead.

(1) Melt the metals together under a little charcoal at a low temperature, and allow the crucible to stand on the furnace-top until the redness has quite disappeared. Pour the alloy, being careful to keep the mould steady until the metal has set.

(2) Repeat, weigh the ingot, which should come out to weight. The surfaces should be quite clean and free from oxidation. If not, remelt and try again. The tin-rich alloy has a brighter appearance than the lead-rich one.

*Exp. 113.* (1) Cut pieces from the ends of the ingots and hammer them into rectangular blocks about  $\frac{1}{2}$  in. long and  $\frac{1}{4}$  in. square. Place these, one at each end of a copper strip about 9 in. long and  $\frac{1}{8}$  in. thick, so supported that it can be heated exactly in the middle by a Bunsen flame. The heat travels equally in each direction. Note the readiness with which the tin-rich alloy melts compared with the other.

(2) Melt the remainder of the lead-rich ingot in a roasting dish over the round Bunsen ; lift the dish on to a brick or iron plate, and stir the metal with a glass rod until it sets, and note its pasty nature during solidification.

**Type Metal.** The two requisites for this alloy are that it shall give a clear, distinct casting, and that it shall be hard enough to stand to its work. These are furnished by an alloy of antimony and tin. Lead with less than 15% of tin antimony expands on cooling and gives it a hard texture. The best kind of the metal lacks

hardness. By the introduction of tin and more than 15 per cent. antimony, a hard compound of Sn-Sb is formed which, distributed through the mass, gives it the necessary hardness.

The usual composition is 60 per cent. Pb, 30 per cent. Sb, and 10 per cent. Sn.

*Exp. 114.* Melt together 24 grams of lead and 4 grams of tin with a little charcoal, and at a low red heat add 12 grams of antimony. Leave for a minute for the antimony to melt, stir well, and pour into a strip mould, the surface of which has been carefully cleaned. Note the bright clear surface of the cast.

Britannia metal is a tin-rich antimony alloy containing a little copper. It is only used for special work at the present price of tin.

**Anti-friction Metals.** The white metals which are used as liners for machinery bearings are known as anti-friction metals. These alloys should possess enough plasticity to adapt themselves to the shaft or axle revolving within, and be sufficiently hard to withstand ordinary wear for a reasonable time. Theoretically, in a well-lubricated bearing, the friction is between the solid parts and the liquid layer of lubricant between them. This would mean little or no wear, but practically this is not so, and the best results are obtained with alloys having the necessary plasticity for easy running, and hard enough to resist ordinary wear. The structure most suited for the purpose is one which consists of a matrix more or less plastic in which are embedded harder particles. The softer parts wear away, leaving the hard particles to carry the load. In this way the area of the rubbing surface is reduced, and it becomes harder and better able to resist wear. The liner is usually contained in a gun-metal or brass shell which

is in contact with the cast-iron housing that supports the shaft.

But another kind of bearing metal has come into use of late years, in which the matrix is hard and is interspersed with soft particles. These are the so-called plastic bronzes which contain lead as an essential constituent. They are largely used for railway work in the United States of America.

The cheapest kinds of bearing metal contain lead and antimony only, but they do not work very satisfactorily. Lead and antimony form a eutectic alloy with 87 per cent. lead and 13 per cent. antimony. With larger percentages of the latter the structure consists of the plastic eutectic interspersed with hard particles of antimony crystals. The necessary structure is there, but the alloy is much better if tin forms a third constituent. This is no doubt due to the fact that tin and antimony form a compound, the particles of which have better wearing properties than the antimony crystals. These are disseminated through the matrix and furnish the wearing surface. In some alloys tin forms the base and is hardened by compounds of tin-antimony and tin-copper, but the price of the principal metal is against their use to any extent.

The phosphor bronzes already mentioned as containing up to 2 per cent. phosphorus are largely used for heavy machinery. Bearings of this class, when accurately made and properly lubricated, give excellent results although they are non-plastic.

*Exp. 115.* Melt 34.8 grams of lead with a little charcoal, and when at a moderate red heat, drop in 5.2 grams of antimony. Leave in the fire for a minute, then stir or shake well, allow to

cool a little, and pour into the rectangular mould. Weigh the ingot.

*Exp. 116.* Repeat, with 32 grams of lead and 8 grams of antimony.

*Exp. 117.* Melt 31.2 grams of lead and 2.4 grams of tin together; add 6.4 grams of antimony and proceed as above.

These three ingots should be broken and their fractured surfaces compared. The first alloy has the eutectic composition, and shows a very uniform structure. The second has a coarser grain, and is harder than either the first or the third. But they are all plastic, and give way under pressure. They may be further tested by rolling.

*Exp. 118.* Remelt the last three alloys and pour them separately into the strip mould. Shear the strips across the middle, so as to get three exactly similar pieces. Pass these through the rolls one after the other with the same pinch. Thus the pieces, being of the same thickness, will be subjected to the same pressure. Repeat the rolling, increasing the pinch with each pass, until the best piece begins to crack. Examine the strips and report on their condition.

The alloy containing tin, although it is not as plastic as the eutectic alloy, is distinctly better than the one containing an excess of antimony, and is much more serviceable for general use.

Another white alloy which is used for the bearings of heavy machinery, such as rock breakers, contains zinc as the principal metal, together with tin and antimony. It possesses considerable compressive strength, and is selected on this account, although its anti-friction properties are rather low.

*Exp. 119.* Melt together 18 grams of zinc and 9 grams of tin with a little charcoal, and add 3 grams of antimony. Leave for a minute at a moderate temperature, then stir well and pour into the strip mould. Grip the strip in

the vice about half-way down, and hammer it carefully till it breaks. Note if it shows signs of bending, and then pass it through the rolls several times with light pinches. Its plasticity is much less than that of the lead alloys.

The lead in the plastic bronzes referred to above tends to segregate in comparatively large patches, and this must be avoided for good work. It is found that the presence of a small percentage of nickel helps to keep the lead more uniformly distributed through the mass. Fairly rapid cooling acts similarly.

*Exp. 120.* Melt together 26 grams of copper and 1 gram of cupro-nickel, 30 per cent. Ni, under a little charcoal. Add, 9 grams of lead and 2 grams of tin. Well stir to ensure that the metals are thoroughly mixed, and pour into a cold rectangular mould. Weigh the ingot ; grip it in the vice about a quarter the length of the ingot from the end, and break it carefully. Remelt the larger piece, and pour it into the strip mould. Pass the strip through the rolls with moderate pinches, and note that the alloy is moderately plastic under pressure. Note the generally uniform structure of the fractured surface of the ingot, and test it under the file. Calculate the percentage composition of the alloy.

**Aluminium Alloys.** Copper, iron, nickel, antimony, and tin all form chemical compounds with aluminium, which, being brittle and crystallizing in the matrix of the alloys, render them of very limited usefulness. However, small percentages of these metals are often introduced for special purposes. On the other hand, zinc, magnesium, and manganese form solid solutions with aluminium, and a series of very useful alloys results. Zinc is the principal addition metal in the light, strong alloys used for motor work. Magnesium is a constituent of "magnalium," but its use is somewhat prohibited on account of price. Some alloys contain copper, tin, lead, and even nickel in small

quantities, but their particular influence on the properties of the alloys is doubtful. Aluminium-zinc alloys containing less than 15 per cent. zinc possess good working properties, and can be rolled or drawn readily in the cold with occasional annealing at a very low red heat. They finish with a good surface under the turning tool and give a well-cut screw thread. With more than 15 per cent. zinc the alloys are hard, but are suitable for castings, which can be readily dressed.

*Exp. 121.* Melt 13.5 grams of aluminium in a clay crucible without charcoal and with the lid on ; add 1.5 grams of zinc, and well stir with a carbon rod or a thin iron poker. Pour into the strip mould. Weigh the strip and residue. Repeat, using 12 grams of aluminium and 5 grams of zinc. Shear two pieces of the same size from the strips, and pass them through the rolls one after the other, at the same pinch, until the softer one shows signs of cracking on the edge. Note that they are very hard. Anneal the softer one for 5 minutes at a low red heat. It is now much softer, and the rolling may be continued.

The melting and casting of aluminium alloys is not easy as it is not advisable to use charcoal. The result is that a very thin skin of oxide forms on the surface of the metal, and this, together with the fact that the molten alloy is somewhat pasty, makes it difficult to pour. The lightness of the metal is also against it, especially with narrow moulds. The top part of the crucible should be hot to prevent chilling, especially in the case of small quantities. A piece of solid zinc chloride, the size of a small nut, may be dropped on to the alloy when it is molten and almost ready to pour. This, if stirred in, is said to assist the pouring of the metal.

**Fluxes for Brass and other Alloys.** Fluxes are sometimes recommended to assist in the melting of metals

and in the preparation of alloys, but considerable experience is required, and care must be taken in their selection. The operator should have a knowledge of the materials he is using, and should avoid complex fluxes unless he is well acquainted with their action. Fluxes should be used sparingly.

Charcoal is not a flux, but it makes an excellent cover for most metals during melting and when molten. It excludes the oxygen of the air, and seldom exerts any harmful effect upon the metal. It should, however, be avoided with aluminium. The best form to use is the coarse powder free from dust.

In melting copper to make ordinary strip castings of brass, bronze, and nickel silver for rolling, it is usual to add a little common salt to the metal just before it runs down. The chloride is said to reduce the cuprous oxide formed in the melting, and so reduce the waste of zinc.

A mixture of Chili saltpetre,  $\text{NaNO}_3$ , and black oxide of manganese,  $\text{MnO}_2$ , is sometimes added to the crucible charge for nickel silver. The saltpetre is said not to oxidize the metal, but actually to reduce any oxide already formed. Also that manganese passes into the metal and increases its soundness.

A mixture of equal parts of calcium sulphate and saltpetre is recommended as a flux for nickel alloys.

A mixture of 3 parts lime and 1 part fluor-spar is said to be a good flux for nickel. The lime is made into a paste, the fluor-spar stirred in, and the mass allowed to set. When broken up it is ready for use.

Yellow prussiate of potash,  $\text{K}_4\text{FeC}_6\text{N}_6$ , may be used as a flux for copper alone. It promotes soundness in the castings.

*Exp. 122.* Melt 40 grams of copper in a clay crucible with the lid on, and when the metal is just melted, add 2 grams of yellow prussiate. Replace the lid, and leave for a minute or two. Pour the metal into a rectangular mould, break the ingot and examine the fracture. It should be close grained, and of a good colour.

*Exp. 123.* Make an ingot of 70 to 30 brass to weigh 40 grams. When the copper is nearly melted, add 2 grams of common salt, and close the furnace to finish the melting. Add the zinc in the usual manner, and cast. Compare the ingot with the one made in Exp. 91. Remelt the ingot, add 5 gram of zinc, and cast in the strip mould. Prepare wires as described in (80), and test them according to the instructions given in (81). Straighten the wires by tapping them on a wood block with the hammer-handle.

A flux must be used in melting brass dust and other finely divided scrap. Calcium sulphate is a very efficient one for the purpose. A small proportion of saltpetre may be added to help the oxidation of impurities.

*Exp. 124.* Weigh 50 grams of brass dust, transfer to a clay crucible, add 3 grams of plaster of paris, and run down in a good fire. Pour the metal into a rectangular mould. Weigh the ingot and break it. Repeat the experiment, using 1 gram of saltpetre in addition.

Ingot metal obtained from brass dust and similar material is always of uncertain composition, and should not be used for strip casting on this account. It is suitable for castings to pattern which have to be dressed only, and which are not to be put to a severe test when in use.

## CHAPTER VIII

### IRON AND STEEL

THE varieties of commercial iron and steel are very numerous and are, perhaps, the most important materials used in manufacturing processes. Commercial iron is the most impure metal on the market, and some, at least, of its useful properties are due to the presence of impurities. The common constituents are carbon, silicon, manganese, phosphorus, and sulphur ; and the different properties exhibited by wrought-iron, steel, and cast-iron are due to the proportions, chemical relations, and distribution of these elements, or their compounds with each other and with iron itself. Such properties are fusibility, malleability, ductility, hardness, tensile and transverse strength, and the properties of being forged, welded, turned, screwed, pressed, etc.

Carbon, silicon, phosphorus, and sulphur all form compounds with iron and manganese, and it is these compounds which modify the properties of the pure iron, or ferrite as it is sometimes called. It would serve no useful purpose to consider the effects of these elements separately upon the metal, as two or more of them are always present in commercial iron. It is therefore the combined effect which is most important ; but we may consider the general effects of the addition of the various elements to metals of known composition.

The materials used in the following experiments are "dead mild" ingot iron and "washed" cast-iron. The former contains only small percentages of the elements, and the latter 3.5 per cent. carbon and still smaller percentages of silicon, etc.

*Exp. 125.* Weigh two lots of 25 grams each of "dead mild" sheet cut into pieces about .5 in. on the side. Pack the pieces loosely into two small clay crucibles, and add enough powdered charcoal to one to well cover the metal. Lute on the lids with moist fire-clay, and place the crucibles side by side, but well surrounded with coke, in a good fire; cover with coke, close the furnace, and leave for an hour. Remove the crucibles and lay them on their sides on the furnace plate; allow to cool; knock off the lids; weigh and examine the contents.

The metal without charcoal is either unaltered or shows slight signs of fusion on the edges; the metal with the charcoal has run down into a button which, when fractured, shows a greyish-white crystalline or granular fracture. The appearance of the fractured surface depends upon the percentage of carbon absorbed from the charcoal, and upon the rate of cooling. It is thus shown that the temperature of the furnace is below the melting-point of ingot iron.

*Exp. 126.* Make a mixture of 6 grams of sifted silica sand (80-sieve) and 6 grams of charcoal. Mix this in a clay crucible with 25 grams of dead mild metal cut into small pieces. Cover with charcoal powder, lute on the lid, and heat strongly for an hour. Withdraw from the fire, allow to cool on the side, fracture and examine the specimen obtained.

A high temperature is required to produce the effect aimed at, as silicon must be reduced from the silica, in order to pass into the iron, along with carbon. If sufficient silicon passes into the metal the fractured surface will be grey, due to the separation of carbon

as graphite when the metal solidifies. Compare with the button obtained in Exp. 125. The button containing the silicon is greyer and easier to file.

*Exp. 127.* Repeat (126) but use 4 grams of powdered black oxide of manganese ( $MnO_2$ ) in place of the silica sand. The metal has a whiter fracture, and often a mottled appearance. It is soft to the file, but harder than (126). The manganese which is reduced from the oxide passes into the metal and tends to keep some of the carbon in the combined form.

*Exp. 128.* Pack loosely 30 grams of dead mild sheet in a clay crucible with the surfaces of the pieces vertical. Place in a good fire, cover with a lid, and then with a thin layer of coke. When the crucible is nearly white hot, shoot in 1 gram of red phosphorus from an iron shoot, and quickly replace the lid. When the phosphorus appears to have burnt off, add a second and a third amount with the same precautions. Close the furnace for a few minutes. When the metal is melted pour it into a warm, round mould. Break the button when cold, and observe the white, close-grained appearance of the fractured surface.

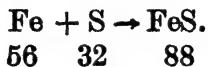
The presence of phosphorus lowers the melting-point of the metal and renders it hard and brittle. The phosphorus combines with some of the iron forming  $Fe_3P$ , phosphide of iron, which dissolves in the excess of iron. The product usually contains about 5 per cent. P.

*Exp. 129.* Repeat (128), but shoot in three portions of 8 grams of coarsely powdered sulphur in place of the red phosphorus. When the mass appears to be molten, make a thin iron poker red hot at the tip, and stir up the molten sulphide. In this way it is easy to determine if all the solid has disappeared; if not add a little more sulphur; otherwise, pour the melt into a round mould. Fracture when cold and examine.

The product is a definite compound of iron and sulphur having the composition expressed by the

formula  $\text{FeS}$ . It has neither the appearance nor the properties of iron.

The reaction is expressed thus :



*Exp. 130.* Loosely pack 25 grams of "dead mild" sheet in a clay crucible. Weigh 5 grams of the iron phosphide from (128) in small pieces. Drop these among the pieces of iron so that they lodge there, and do not fall to the bottom of the crucible. Well cover with charcoal powder; lute on a lid, and heat strongly for an hour. Take the crucible from the fire, and lay it on its side to cool. Fracture the button, and compare the fractured surface with (125). The effect of the phosphorus is to keep the carbon in the form of carbide as is shown by the whiter fracture of the sample.

Q. Assuming that the phosphide used contains 5 per cent. of phosphorus, what is the percentage of the element in the button obtained?

*Exp. 131.* Roughly crush a little of the iron sulphide made in (129); weigh 1 gram in small pieces, and repeat (130) with the sulphide in place of the phosphide. The success of the experiment depends largely upon the melting of the sulphide on the surface of the hot iron. Fracture the button and compare the surface with that of (125). The colour is lighter and the fracture usually exposes one or more blow-holes. The metal is not sound.

### Carburizing Cementation and Case Hardening

This process consists in the gradual absorption of carbon by iron or mild steel while in the solid state and at a temperature of from  $900^{\circ}$  C. to  $950^{\circ}$  C. A hard steel shell is thus formed round a tough iron core. The thickness of the shell and the concentration of carbon in it depend upon the temperature, the dura-

tion of the heating, and the activity of the carburizing mixture. A large number of mixtures are used commercially, but the general principles of the process will be best illustrated by the use of one or two simple mixtures. Thus scrap leather thoroughly charred out of contact with air produces a carbonaceous mass much used for the process. Also wood charcoal crushed and sifted through a 10-sieve, and then through a 40-sieve to remove the dust, works excellently when well mixed with one-tenth its weight of dry sodium carbonate (soda ash). Iron boxes closed with a lid are used for conversion on the large scale, but a clay crucible may be employed for experimental work.

*Exp. 132.* Fill a clay crucible with the wood charcoal mixture and embed in it two pieces of  $\frac{3}{8} \times 1\frac{1}{2}$  in. round iron or mild steel rod, and two pieces of  $\frac{1}{16}$  in. sheet iron. See that each piece is completely surrounded by the mixture. Put on a closely fitting lid, and place the crucible in a gas muffle, the temperature of which is about  $930^{\circ}\text{C}$ , *i.e.* a good red heat. Close the muffle and leave for  $1\frac{1}{2}$  to 2 hours. Take out the crucible, remove the lid, and water-quench one of the pieces of round bar without disturbing the others. Replace the lid and allow the crucible to cool.

The experiment may be repeated by using the charred-leather composition, and a comparison of results thus obtained.

*Exp. 133.* Take the round bar and one of the pieces of sheet metal which were allowed to cool in the crucible, and dip them into a thin paste of borax and water, dry them on a hot plate, and place them on a clean fire-clay slab in a moderately hot muffle (about  $800^{\circ}\text{C}$ ). When red hot, remove the pieces and water-quench them rapidly. The function of the borax is to retard the oxidation of the metal during reheating.

*Exp. 134.* Fracture the two bars obtained above by a sudden blow, when the piece is gripped about one-third of its length

in the vice. Note the shell of altered metal and the core. The core of the bar which was quenched without reheating is not so uniform in structure as that of the reheated bar. This makes it clear that case-hardened work, which is allowed to cool and then reheated before quenching, is more reliable than that quenched straight from the pot. Next fracture the two pieces of sheet metal. The reheated one breaks off short, and shows a hard steel through fracture ; while the slowly cooled one bends somewhat before it breaks.

*Exp. 135.* Take four pieces of rod iron as described in (132), and embed them in charcoal mixture as described above. Heat in a muffle for  $1\frac{1}{2}$  to 2 hours ; remove and allow to cool. Take out one of the bars and prepare the pot for a second heating. Repeat the process a third and fourth time. Note the time that each piece has been exposed to the carburizing action, and when the experiment is finished prepare the rods for reheating as described above ; reheat and water-quench. The rods may then be fractured and their fractured surfaces compared.

This experiment is a very instructive one as showing the gradual nature of the absorption of carbon by the metal ; but when carried out by students with limited time at their disposal must extend over several weeks. If the muffle space is limited, two or even four students may work with one pot, and share the results. A student working all day may proceed more rapidly. In that case the pot need not be allowed to cool down after each heating, but the red-hot bar, when removed, should be dropped into a crucible containing hot charcoal to prevent oxidation during cooling, and the pot put back into the muffle at once. Time is thus saved.

*Exp. 136.* Cut off four pieces of well-drawn iron wire 6 in. long and about  $\frac{1}{8}$  in. diameter. Straighten the wires by tapping them on the bench with the hammer-handle to prevent bruising the metal. Fit a clay plug into the end of a piece of strong iron tube about 8 in. long and 1 in. internal diameter. Stand the tube upright, and fill it with charcoal mixture ; then carefully insert the four

wires so that each is surrounded by the mixture, and force in a stiff clay plug to meet the ends of the wires so as to keep them in position. Place the tube in a hot muffle and heat it for two hours. Remove, and allow to cool. Reserve the cemented wires for further experiments.

**Tool Steel.** This variety of steel is usually made in crucibles in this country, although the electric furnace is gradually being introduced into the industry. The original method was to cement bar-iron, cut up the blister bars so produced, and melt the pieces in crucibles. This method is still used for some varieties of steel, but quicker and cheaper methods are now more largely adopted, and excellent material is produced. It is shown in Exp. 125 that when wrought-iron is strongly heated with excess of charcoal the metal runs down into a button. The product is cast-iron, but if the quantity of charcoal is limited to about that required to produce steel with a given content of carbon, then steel is the result. This is one of the methods adopted. Also, if bar-iron and "washed" iron are mixed in proportions to give the proper carbon content, and the mixture is melted, steel is the result. Mild steel scrap is also mixed with "washed" iron for the same purpose. In all cases, however, it is necessary to add ferro-manganese to the melt to *kill* it and give sound castings. Aluminium is also used for the same purpose.

*Exp. 137.* Fill a clay crucible with charcoal powder, and embed in it 20 grams of thin sheet iron, cut into pieces of suitable size. Cover the crucible with a lid and heat it for 2 hours in a hot muffle. Allow the crucible to cool before removing the cemented metal. It will have absorbed less than 1.5 per cent. of carbon.

*Exp. 138.* Cut the cemented metal into small pieces and

transfer it to a clay crucible ; put with it 1 gram of ferro-manganese in small pieces and 1 gram of charcoal. Sprinkle 2 grams of borax over the metal, and lute on a lid. Place the crucible in a good position in the fire, and cover it with coke. Close the furnace, and leave it for an hour or more, but put in more coke if the fire burns down too rapidly, and work it under the crucible. Remove the pot from the furnace, and place it on its side to cool. Break away the lid and examine the contents of the pot.

If the metal has properly melted, it will be in the form of an oval button lying in a bed of slag, from which it can be chipped out, or the crucible broken to remove it. The button should be gripped near one end in the vice, nicked with a chisel, and broken to expose the fracture, which should have the close-grained structure of steel. The furnace must work at its best to obtain the result ; it is much easier if a salamander crucible is used, but more carbon passes in, and the metal is nearly, if not quite, pure white iron.

*Exp. 139.* Put 15 grams of sheet iron, cut into small pieces, into a clay crucible, add 10 grams of white cast-iron in small pieces and 1 gram of ferro-manganese. Sprinkle 2 grams of borax over the mixture, lute on a lid, and proceed exactly as described in the last experiment.

Assuming that the "washed" iron contains 3 per cent. and the sheet iron .05 per cent. of carbon, 10 grams of the former would contain .3 gram, and 15 grams of the latter .0075 gram of carbon. As these quantities are distributed through the 25 grams of steel its content of carbon will be  $.3075 \times 4 = 1.23$  per cent. The actual content is usually lower than that calculated.

*Exp. 140.* Heat the larger piece of either of the steel buttons obtained above to a bright red heat and forge it into a square bar. It will have to be reheated two or three times in the process. Nick the bar, break it, and examine the fractured

surface. Test one of the pieces with a file; reheat the other piece to a moderate red heat and quench it in water. Test also with the file.

The experiments just described give a general idea of the manufacturing processes for introducing carbon into iron, but no flux is used on the large scale, and the ferro-manganese is added to the molten charge.

**Hardening and Tempering.** Carbon exists in two forms in iron and steel: (1) In the free state as graphite, which is distributed through the mass of the metal in the form of flakes or small particles, as in grey cast-iron; (2) in the combined form, when it is in combination with iron and manganese. It is then known as *carbide carbon*. The whole of the carbon in wrought-iron and steel is in this form under normal conditions, and in ordinary steels it is the relation of this carbide carbon to the ground mass that confers upon the metal its characteristic properties after subjection to heat treatment. Thus a piece of steel when properly annealed or normalized, and therefore in its softest condition, has its carbon wholly in the form of free carbide; that is, as a definite constituent of its structure. On heating the metal to a temperature above 700° C. the whole of this carbide passes into solid solution, and thus ceases to be a separate constituent of the mass. The solid solution is hard, and if the steel is suddenly quenched from a temperature above 700° C. the solution remains intact, and the quenched product is glass-hard and brittle. The more sudden the quenching the harder and more brittle is the steel. The brittleness is in part at least due to strains set up in the mass, caused by unequal cooling of the parts, but the hardness is a property of the solid solution.

On the other hand, if this hard and brittle metal is gradually raised in temperature, the carbide begins to separate, and the strains are removed. Thus the metal becomes softer and less brittle. This action is known as tempering, and its extent depends upon the temperature to which the metal is raised. In ordinary practice it seldom exceeds 290° C.

*Exp. 141.* Take two pieces of sheet steel (spring steel) 3 in. by  $\frac{3}{8}$  in. wide. Place them in a hot muffle, and, when they are red hot, quench one in oil and the other in water. Test the pieces separately with a file, and note difference in hardness. Fix one of the pieces in a vice about  $\frac{1}{2}$  in. from one end ; hold the other end in the pliers and bend it over. Note the angle at which it fractures. Repeat with the other piece. Take the longer pieces, and brighten one side of the water-quenched specimen by rubbing on an emery pad. Place it on an iron bar at a low red heat with the bright side up, and watch the play of colour over the surface. When it becomes light blue tip it into water to quench it. Dip the other piece into oil, and allow the excess of oil to drain away ; then ignite the oil on the surface in a Bunsen flame, and as soon as it has burnt off, quench as before in water. Test with file and in the vice, and note that the strips are softer and less brittle than before tempering.

The smithy hearth and the muffle furnace, although much used for hardening purposes, are often replaced by the lead bath, or the salt bath, which for some purposes gives better results, and in addition prevents oxidation of the metal, except at the surface of the bath, when the tool is only partly immersed. But as this oxidation takes place over a very small surface at the junction, it is usually of no importance. Of these various furnaces, the first is the least and the last the most under control. The salt bath, fitted with a pyrometer to register its temperature, gives the most accurate hardening results possible. Also by using a

mixture of salts in the bath any special temperature can readily be obtained and special work carried through. The last two methods may easily be illustrated on the small scale.

*Exp. 142.* Arrange a small gas furnace for heating lead in a clay crucible. The Méker furnace is very suitable for the purpose. Add lead to the crucible till it is nearly full of the molten metal, and continue the heating until the contents are at a moderate red heat. Add just sufficient coarsely powdered charcoal to form a thin layer on the surface of the metal to protect it from too rapid oxidization. Brighten a piece of  $\frac{1}{4}$  in. soft steel rod about 4 in. long with a smooth file or with emery cloth ; grip it in a pair of tongs, and immerse it about half-way in the red-hot lead. It will have to be held in position as the metal is much lighter than the lead, and would float to the surface if not held down. When the steel has reached the temperature of the bath, remove it and water-quench as before. Test the two ends with a file. The end not heated will be unchanged, and will respond to the file as it did before the experiment.

*Exp. 143.* Nearly fill a plumbago crucible with dry common salt, and arrange it in the Méker furnace as above. When the salt is melted add more until a convenient quantity of the liquid has been obtained. Repeat the operations described in (142) ; but it will not be necessary to hold the steel down as the salt is much lighter than the metal. Compare the results of the two experiments.

If a thermo-junction, protected by a clay tube and connected with a direct reading milli-voltmeter, is inserted in the molten metal, or salt, as the case may be, the temperature of the bath can be read and any variation in its value noted. Lead melts at  $328^{\circ}$  C., but the bath may be raised to any temperature above this within the limits of the furnace. Common salt melts at  $795^{\circ}$ – $800^{\circ}$  C., and the bath is hot enough for ordinary work. For special work salt mixtures having known melting-points are sometimes used, and the

steel may be raised to a few degrees above a minimum quenching temperature. It will be noticed that when a cold tool is put into the molten salt a layer of the solid salt is formed on the surface of the metal. This layer, on account of the low conductivity of the salt for heat, remains solid until the metal reaches the melting-point of the salt. Thus, if the rod is withdrawn and its surface is covered with the fused salt, it is at the temperature of the bath. If then it is held until the thin liquid layer on its surface just solidifies, it is at the freezing temperature of the salt, and may be quenched.

**Pyrometry.** It is outside the scope of this book to describe at length the various forms of pyrometers, but the student interested in the heat treatment of metals should consult special works on this important subject.

The electrical pyrometer, briefly indicated above, consists of two wires, of platinum and 10 per cent. rhodium-platinum, twisted together at one end. The free ends are connected with recording apparatus, by which the feeble currents, generated when the junction is heated, are measured and the indications registered in degrees of temperature. Some recorders are automatic, and give temperature curves over considerable ranges.

The absorption of light coming from the heated space or material, by means of coloured glass or liquid, is also largely used for determining temperature. The instruments are called optical pyrometers, and are direct reading.

The softening of clay mixtures (Seger cones) and the melting of salt mixtures (sentinel pyrometers) are also used to indicate approximately the temperature of

furnace spaces. If two sentinels, one of which melts at  $800^{\circ}$  and the other at  $820^{\circ}$ , are put into a furnace, and the first melts, while the other remains intact, the temperature of the space is between  $800^{\circ}$  and  $820^{\circ}$ .

Technical skill, which is only obtained by experience, may be assisted by the use of pyrometers, but to rely upon them as an entire substitute for the human eye and judgment is not to be recommended, and may result in failure.

**Alloy Steels.** For some purposes carbon steels have been superseded by self-hardening and high-speed steels. These are iron alloys containing, in addition to carbon, two or more of the following metals: manganese, tungsten, molybdenum, and vanadium. While chromium, nickel, and quite recently cobalt, are constituents of steels having a high resistance to shock, and great tensile strength. It will be instructive for the student to make some experiments with these steels, and thus become acquainted with the materials, such as ferro-tungsten, ferro-chrome, and similar alloys, used in the manufacture.

The self-hardening steel, which contains manganese and tungsten, was first made for use by Mushet, and is still known by his name. If a tool made of this steel is raised to an orange heat, and allowed to cool naturally, it is ready for work. The self-hardening property is due to the presence of sufficient manganese, while the function of the tungsten is to keep the carbon in the hardening form at temperatures at which a carbon steel would soften and become useless.

*Exp. 144.* Make a 20-gram sample of self-hardening steel containing 2 per cent. carbon, 2.5 per cent. manganese, 5 per cent. tungsten and 1 per cent. silicon. Weigh 9.5 grams of

cemented sheet in small pieces, .6 gram of 80 per cent. ferro-manganese, 3.6 grams of 28 per cent. ferro-tungsten, and .8 gram of 25 per cent. ferro-silicon. Transfer the materials to a clay crucible, sprinkle 2 grams of borax over the mixture, lute on a lid, and heat the crucible as strongly as possible for 1½ hours. Proceed as described in Exp. 138.

This experiment is not an easy one, and much depends on the care bestowed upon the firing. A good position should be selected in the fire, not too low down, and the crucible should be kept from sinking too much by working the fire under it when adding more coke. The fractured surface of the button shows a close-grained structure, and the metal is hard to the file.

High-speed steels are similar in composition, but contain chromium and less manganese. In some brands molybdenum takes the place of tungsten.

*Exp. 145.* Make a 20-gram sample of high-speed steel containing 1.5 per cent. carbon, .5 per cent. manganese, 2 per cent. chromium, and 6 per cent. tungsten. Weigh 94.4 grams of cemented sheet, .6 gram of ferro-manganese, .7 gram of 60 per cent. ferro-chrome, and 4.3 grams of 28 per cent. ferro-tungsten. Proceed as in the last experiment.

The button of steel obtained is very hard and difficult to break. The hardness is increased by raising it to as high a temperature as possible and then directing a blast of air upon it by means of the foot-blower.

### Cast-iron

Pig- and cast-irons vary considerably in composition, though the constituents are practically always the same. They are: iron, carbon, silicon, manganese, phosphorus, and sulphur, but the proportion of the constituents other than iron may vary from 9 per

cent. in a grey phosphoric pig to 4 per cent. in a good white iron. Carbon is present both as graphite and as combined, or carbide carbon, and the ratio of the two determines the character of the fractured surface and other properties of the metal. Thus, when the carbon is nearly all in the form of graphite the fracture is grey and open, and the metal is soft ; when combined, the fracture is white, and the metal hard and brittle. The total carbon may vary from 2-4 per cent.

The character of pig-iron depends largely upon the percentage of silicon present, which varies from 2-3.5 per cent. ; and it may be taken generally that, the higher the silicon content, the greyer the iron will be. Therefore silicon in its relation to carbon is a softener, as it tends to separate carbon from its combination with iron. Manganese has an opposite tendency to that of silicon, and also serves as an antidote for sulphur. Phosphorus tends to harden and weaken the metal, and also to render it more fluid when molten. The percentage of phosphorus in ordinary foundry iron usually varies from 5-1.5 per cent., which may be increased to 2 per cent. for ornamental castings requiring a fine skin. Sulphur is to be avoided as much as possible, but may be present from 0.5-1 per cent.

The experiments which follow are intended to illustrate the general influence of the common constituents of cast-iron upon its properties. "Washed-iron," a pure white iron, containing very little but iron and carbon, forms an excellent bulk material into which the other constituents of cast-iron may be introduced. It is referred to in the instructions as white iron.

The experiments are best made in a Morgan salamander crucible, size 1/0. The materials are carefully

weighed, and the white iron is placed at the bottom of the crucible ; the other constituents of the mixture are arranged on the top. Powdered material should be avoided as much as possible, as the powder tends to work down to the bottom of the crucible and often does not melt in properly. The charge is lightly covered with powdered charcoal. Too much charcoal prevents the molten metal from leaving the crucible freely, and a bad cast is the result. The most convenient form for the casting is a rod about  $\frac{5}{8}$  in. by 2 in. This is cast in a sand mould, for which an iron mortar about 4 in. deep makes a suitable flask. The pattern, a smooth wooden rod about 4 in. long, is held upright in the mortar, upon the bottom of which some moist casting sand has been rammed, and more sand is rammed round it to the depth of  $2\frac{1}{2}$  in. The rod is then carefully withdrawn and the edge of the mould bevelled with the finger. Finally the rod is rubbed over with powdered charcoal, worked into the mould, and turned round. This gives the casting a better surface than when the molten metal comes into direct contact with the raw sand. When the metal is ready, take the pot from the fire and pour it quickly, as small charges chill rapidly when removed from the fire. When cold, the rod can be broken in the vice and the fractured surface examined.

*Exp. 146.* Melt 100 grams of white iron and cast it in a sand mould. Allow to cool ; fracture the rod and note the blow holes in the section. A pure cast-iron practically free from silicon does not give a sound casting, this being due to the liberation of dissolved gases when the metal solidifies.

**Silicon.** This element, in addition to acting as a softener by setting some of the carbon free, causes the

metal to give sound cast. It is added in the form of ferro-silicon, which is made in several grades. One containing about 25 per cent. Si serves very well for experimental work. The weight of alloy required is found from :

$$\frac{\text{Silicon per cent. required}}{\text{Silicon per cent. in the ferro-silicon}} \times 100.$$

This weight is made up to 100 grams with white iron.

*Exp. 147.* Make a mixture of white iron and ferro-silicon to contain 1 per cent. of silicon. Melt and cast in a rod mould. If the ferro-silicon contains 25 per cent. silicon then  $\frac{1}{25} \times 100 = 4$  grams = weight of the ferro alloy to be added to 96 grams of white iron.

*Exp. 148.* Make similar mixtures containing 2 and 3.5 per cent. silicon respectively. Melt and cast. Allow the rods to cool in the sand.

The rods should be broken in the vice near to the bottom end of the casting, and the fractured surfaces examined. They should also be tested for hardness with a file. Greyness and softness increase with the increase in the content of silicon. Although silicon readily alloys with iron and hardens the metal, it also causes combined carbon to separate as graphite; and as the hardening effect of silicon is less than that of combined carbon the metal is generally softened. This is the simple explanation of the above results.

**Manganese.** This metal also combines with carbon to form a carbide,  $Mn_3C$ , corresponding to the iron carbide,  $Fe_3C$ . If then manganese is present in a cast-iron, it shares the combined carbon with the iron, and influences the general result. Graphite is not so readily set free from the manganese carbide as from the iron compound; consequently a cast-iron containing

manganese is closer grained and harder than one of the same silicon and carbon content, but free from the metal. Ferro-manganese, which may contain upwards of 80 per cent. manganese, is used for introducing the metal into cast-iron. The amount required is calculated in the same way as for ferro-silicon.

*Exp. 149.* Make a mixture of white iron, ferro-silicon, and ferro-manganese to contain 3.5 per cent. silicon, and 2 per cent. manganese. Calculate the weights of the two alloys required, and make up to 100 grams with white iron. Follow the general instructions and run the metal into a rod mould. Break the rod near to the bottom end, and compare the fracture with that of the similar rod made in (148), but containing no manganese. Test with the file.

The structure should be closer in the grain and lighter in colour than the manganese-free rod, and the metal should be distinctly harder under the file.

The other function of manganese, which may be made the subject of experiment, is its effect upon the action of sulphur on the structure and properties of cast-iron. Sulphur should not exceed 1 per cent. unless it is introduced for [a special purpose, or in castings, such as sash weights, the composition of which is of no importance.

*Exp. 150.* Make a 100-gram mixture of white iron, ferro-silicon and iron sulphide to contain 3 per cent. silicon and 1 per cent. sulphur. Iron sulphide contains  $\frac{4}{11}$  its weight of sulphur. The sulphide should be in small pieces, not powder, and should be dropped on to the white iron before the ferro-silicon is added to the mixture in the crucible. When melted pour the metal into a rod mould.

*Exp. 151.* Make a similar mixture but containing in addition 6 per cent. of manganese, and proceed exactly as before.

Excess of both elements is introduced in these experiments in order to make the influence of the

manganese quite distinct. This will be rendered clear from the appearance of the fractured surfaces of the rods.

**Phosphorus.** The general effects of this element on the structure and properties of cast-iron are as follows: the metal is harder, closer grained, and lighter in colour than similar metal practically free from it. It melts more readily and is very fluid, filling the mould and giving a casting with a clear surface.

*Exp. 152.* Make a mixture of white iron, ferro-silicon, and iron phosphide to contain 3.5 per cent. silicon and 1.5 per cent. phosphorus. Take the phosphide as containing 5 per cent. phosphorus. Calculate the weights of ferro-silicon and phosphide required, and make up to 100 grams with white iron. Melt and cast. Compare the fracture with that of a rod of similar composition, but free from phosphorus. *Exp. 148.*

**The Moulding and Casting of Test-bars.** This work is necessary for the student who is making a special study of cast-iron, and if he has a practical knowledge of moulding it is easily carried through. The iron-founder may mould and cast square bars for transverse testing and round bars for tensile tests. He will be able to determine the influence of silicon and manganese on the strength of cast-iron by introducing different proportions of those elements. The arrangement of such work must, however, be left to the demonstrator.

**Malleable Cast-iron.** By proper heat treatment it is possible largely to decompose the iron carbide in white cast-iron, partly to remove the carbon by oxidation, and to deposit most of the remainder in the free state, a little being left as carbide. This free carbon is intermixed with free iron in the structure of the changed metal, and has so little influence upon its properties

that the metal resembles wrought-iron in that it is quite soft and tough. It can be filed, turned, and screwed, and has considerable toughness and tenacity. It differs from wrought-iron in that it cannot be forged or welded. The process is called malleablizing or oxidizing cementation.

*Exp. 153.* Weigh out 4 grams of 25 per cent. ferro-silicon, and 96 grams of white iron. Transfer the mixture to a salamander crucible, and sprinkle a little charcoal over it. Put the crucible into a good fire so as to get a thoroughly fluid melt. Make three rod moulds in sand about  $\frac{3}{8}$  in. diameter and  $2\frac{1}{2}$  in. deep. Stir the metal with a red-hot poker to see that it is quite fluid, and pour it rapidly into the moulds. Remove when cold.

The rods are very hard and brittle, and when broken show a perfectly sound fracture. As "washed" iron is used, the silicon will be a little under 1 per cent. The metal is a good quality white iron.

*Exp. 154.* Place the rods or part of them upright in a clay crucible, and surround them with coarsely powdered red haemite; lightly lute on the lid, and heat the crucible for 2 hours in a muffle at about  $900^{\circ}\text{C}$ . Remove the crucible, and when cold take out one of the rods. Replace the lid, and heat the crucible again for 2 hours; take out the second rod. Repeat with the third rod. The temperature of the muffle must not be too high or the ore will soften and stick to the rods. Test the rods in the vice. Break them carefully with the hammer, and test the fractured surface with the file.

It is probable that the file-test will not distinguish between the bars, but the one that has been longest exposed to the heat treatment should bend somewhat before breaking. The carbon separates from the carbide fairly rapidly at the proper temperature, and the metal is softened, but a longer exposure is necessary if the metal is to be tough as well as soft. On the

manufacturing scale the exposure to the maximum temperature varies from 12-96 hours, according to the size of the castings ; and as several days are occupied in heating up and cooling, the process is a lengthy one. A well-malleabilized piece will bend upon itself before breaking.

**Tensile Tests.** The wires prepared in (136) may be used to illustrate the general influence of carbon on the mechanical properties of the metal. Three wires should be tested as described in (81), and the results set out in the form of a table : (1) an unchanged wire, (2) a cemented wire just as it came from tube, and (3) a cemented wire which has been raised to a red heat and hardened by quenching in oil. It will be found that the percentage elongation decreases, and the tensile strength increases for the three wires in the order given.

## CHAPTER IX

### **GOLD AND SILVER**

THIS chapter is devoted to practical work which should prove useful to those handling the precious metals in the process of manufacture. The pure metals are of little use for this purpose, for although they have excellent working properties, they are too soft to resist wear and tear for any length of time. They must therefore be hardened by the addition of other metals, of which copper is by far the most important. Thus silver coins contain 92.5 per cent. silver and 7.5 per cent. copper, and gold coins 91.6 per cent. gold and 8.3 per cent. copper, or 2 parts of copper in 24 parts of the alloy. Gold alloys used for jewellery contain both copper and silver, with sometimes a fourth metal, such as zinc or iron, but the best working properties are possessed by those alloys from which the fourth metal is absent. The effect of copper is to deepen the colour of gold alloys, and that of silver to lighten it, so that various colour-effects may be obtained in alloys of the same carat by varying the proportions of copper and silver. Other metals are added to impart particular properties, either real or fancied, to special alloys. Such metals as tin, antimony, and lead are harmful, and care should be taken to prevent their entering

alloys which have to be worked much either between the rolls or under the hammer.

**Cupellation.** This process, which is a very ancient one, depends upon the fact that gold and silver do not oxidize when heated in air ; that they alloy readily with lead ; and that the lead can be completely removed by converting it into oxide at a sufficiently high temperature to keep the oxide molten, so that it may be either absorbed or volatilized, and thus removed from the unoxidized gold and silver. The temperature at which the process is conducted is such that the unoxidized metal is quite molten when the last traces of lead are removed. The temperatures required are about  $1000^{\circ}$  C. for silver and  $1100^{\circ}$  C. for gold. Platinum cannot be cupelled in the ordinary way, as its melting-point is much above the muffle temperature. Either the oxy-hydrogen flame, or the electric arc, with a lime bed would have to be used.

The muffle-furnace is required for cupellation experiments and, as rapid oxidation is essential, the back of the muffle-chamber should be perforated by two or three holes towards the top. This encourages a steady current of air through the chamber and accelerates the oxidation.

Cupels for assaying may be made of bone ash, moistened, and strongly pressed in a mould. They should stand for some time in a warm place to dry and temper before being used. Cupels are now made in which Portland cement forms part of the mixture. They are firmer and safer to handle. One mixture contains 5 parts of Portland cement and 1 part of sand.

*Exp. 155.* Arrange three small cupels in a row across a hot muffle. Weigh out 3 grams of lead, 1 gram of thin sheet copper,

and 1 gram of silver. Place the metals separately on the three cupels, and close the muffle for 20 minutes. If the whole of the lead has now disappeared remove the cupels from the muffle, examine them, and weigh the copper and silver.

The lead is completely oxidized and the oxide partly absorbed by the cupel and partly volatilized. The copper is partly oxidized, and has increased in weight, but has not fused. The silver may have fused to a bead, but is the same weight as when put in. It is clear, then, that if copper oxide is to be absorbed by the cupel it must either melt or be dissolved by a molten oxide, which itself sinks into the cupel and carries the dissolved oxide with it. Now lead oxide will dissolve copper oxide to a limited extent and so carry it into the cupel, but at least sixteen times the weight of lead is required.

*Exp. 156.* Place two large cupels in the muffle to get hot, and then weigh .5 gram silver, .5 gram copper, and 9 grams of thin sheet lead. Wrap the silver and copper in the lead and hammer the whole into a packet rather smaller in diameter than the surface of the cupel. Make up a similar packet with .5 gram silver, .5 gram copper, and 4 grams of thin sheet lead. Transfer the packets to the hot cupels, and close the muffle. Note the progress of the cupellation from time to time, and when the silver with the larger quantity of lead brightens, remove the cupels, and take off the beads with the pliers. Weigh the beads and wrap the heavier one in 5 grams of lead ; put it back in the same cupel, and finish the cupellation. Weigh the bead and compare it with the first.

The heavier button is evidently an alloy of silver and copper oxidized on the surface. Therefore there is a lower limit to the quantity of lead required to carry copper into the cupel ; below this, some of the copper is left in the alloy when the whole of the lead

has disappeared. There should not be less than sixteen times as much lead as copper.

**Assay of Lead containing Silver.** If the lead is free from other metals, such as tin and antimony, the assay is effected by simple cupellation. The weight of argentiferous lead to be taken for assay will depend upon the content of silver.

*Exp. 157.* Put two medium cupels into the muffle to heat; weigh two lots of the lead of 10 grams each, hammer them into suitable packets and transfer them to the cupels. When the cupellation is finished, withdraw the cupels and take off the silver beads with the pliers; see that they are clean where they have been in contact with the cupels. Weigh separately. The mean of the two weights multiplied by 10 gives the per cent. silver in the alloy.

*Exp. 158.* Melt together 50 grams of lead and 1 gram of silver in a clay crucible. Add a little charcoal to prevent oxidation, and melt in the wind furnace. When the crucible is red hot, well stir the metal with a charred stick, take the crucible out of the fire, and allow to stand till the redness has disappeared. Pour the alloy into the rectangular mould; weigh the ingot, and roll it into a strip about 9 in. long. Cut the strip into three equal pieces; weigh them to exactly the same weight (about 10 grams). Hammer the pieces up into packets and cupel them on separate cupels. Remove the beads, which should all be the same weight, thus showing that, if the alloy is properly made, it is homogeneous within the limits of the experiment.

Commercial lead contains silver, so that in very accurate assays lead especially free from silver should be employed. The student may assure himself of the presence of silver by cupelling a sample of any ordinary commercial lead.

*Exp. 159.* Place a large cupel in the muffle, and when it is hot transfer to it 40 grams of lead made into a packet not quite as wide as the cupel. When the cupellation is finished, examine the surface of the cupel for traces of silver. The bead is very

small, but can always be seen, especially with a hand glass. It is too small to weigh.

The removal of tin, zinc, and antimony from silver alloys is more difficult than that of copper. The cupels are badly corroded, and the assay is unsatisfactory.

*Exp. 160.* Weigh .5 gram of silver, .25 gram of antimony, and 9 grams of lead. Make into a packet for cupellation. Prepare a similar packet containing .2 gram of tin in place of the antimony. Transfer the packets to two large cupels in the muffle, and leave until the silver appears. Remove the beads, and note the condition of the cupels. Weigh the beads to see if any loss has occurred.

**Scorification.** As it is not possible to remove notable quantities of tin and antimony, or even copper, from poor silver alloys by cupellation, it is necessary to get rid of these metals by a preliminary operation. This is known as scorification, and is carried out on a thick clay dish, known as a scorifier. When lead containing another oxidizable metal is allowed to oxidize freely, the oxide of lead formed acts as an oxidizing agent to the other metal and assists the air in oxidizing it. Thus the second metal oxidizes faster than the lead does, in proportion to the amount present, and is completely oxidized while some of the accompanying lead is still untouched. If the operation is carried out on a clay dish, the oxides formed unite with the silica of the clay and a complex silicate rich in lead, and containing the other metal, is the result. This is the scoria, which is quite fluid and can thus be poured from the dish together with the unchanged lead. When solid, the scoria can be broken away and the metal button separated. Any silver or gold present

in the original alloy is left in the lead, and can be obtained from it by cupellation.

*Exp. 161.* Put a scorifier in the muffle to get hot. Weigh .5 gram of silver, 2.5 grams of copper, and 25 grams of lead. Make them into a packet, and place it on the hot scorifier. Continue the scorification until the molten scoria just covers the metal. Pour the whole into a round mould, and allow it to stand until the metal has set, as the scoria solidifies first; so that, if the mass is turned out of the mould too soon, the lead will splash and loss may occur. Break away the scoria, and hammer the metal button on the edge to see that it is soft. Cupel and weigh the silver bead.

*Exp. 162.* Weigh .5 gram of silver, 1 gram of tin, and 45 grams of lead.

Also .5 gram of silver, 1 gram of antimony, and 30 grams of lead.

Make into two packets, transfer to hot scorifiers, and proceed exactly as in the last experiment.

It will be noticed that tin requires more lead to form a liquid scoria than antimony does. If insufficient lead is used, the tin oxide collects in the solid state on the surface of the liquid and the operation is unsatisfactory. A good muffle temperature is required.

Scorification should always be resorted to as a preliminary to cupellation when hard lead has to be dealt with, and also when much lead has to be removed. The lead is easily recovered from the scoria, but is hard from the presence of the other metals which are reduced with it.

*Exp. 163.* Crush the scoria obtained above in an iron mortar; weigh 20 grams of the powder, and mix with it 20 grams of sodium carbonate. Transfer the mixture to a clay crucible, insert a piece of hoop iron about 3 in. long, and put the crucible into a good fire. When the mass has settled down into steady fusion, stir it with the iron strip, tap the strip on the side of the crucible to dislodge any globules of lead which may adhere to it, and

invert the strip. Allow to remain in the fire for a minute, remove the strip as before, and pour the contents of the crucible into a round mould. When cold, break away the slag, hammer the lead button on the edge, and weigh it.

The important lead ore, galena, always contains silver, and is one of the sources of this metal. The lead compound present in the sulphide is  $PbS$ , and it is accompanied by more or less silver sulphide,  $Ag_2S$ . When the ore is treated in the ordinary process of extraction, the silver is reduced along with the lead and passes into it. The lead then undergoes further treatment for the separation of its silver.

*Exp. 164.* Mix together 20 grams of finely powdered galena, 18 grams of sodium carbonate, and 2 grams of tartar. Transfer the mixture to a clay crucible, cover with 2 grams of sodium carbonate, insert a strip of hoop iron, and proceed exactly as described in the last experiment. Weigh the button of lead obtained, and cupel it. Weigh the silver bead. Calculate the percentage of lead and silver in the ore from the weights of the metals obtained.

It is usual to make two assays of the same ore so that one may serve as a check upon the other. Also another ore with a different content of silver may be assayed. Two or more assays may be made in the same crucible.

Silver ores free from lead may be smelted in admixture with lead oxide and charcoal. The oxide is reduced, and the liberated lead dissolves the silver from the ore. If at the same time the earthy matter is slagged off, the lead separates readily, and the silver may then be obtained from it by cupellation.

*Exp. 165.* Weigh 20 grams of the finely powdered ore, 30 grams of red lead, 2 grams of charcoal, 20 grams of sodium carbonate, and 10 grams of borax. Well mix the red lead and

charcoal, add the ore and mix again; then add the sodium carbonate, transfer the whole to a clay crucible and cover with the borax. The crucible should not be more than two-thirds full. Melt in a good fire, and when tranquil pour into a round mould. Break away the slag, hammer the lead button to see that it is soft, and cupel it. Weigh the bead, and calculate the percentage of silver in the ore. Make two assays.

**Preparation of Pure Silver.** Fine silver always contains traces of copper. For accurate work this should be removed and the metal obtained as near 100 fine as possible. Silver scrap, however, may be used in the preparation.

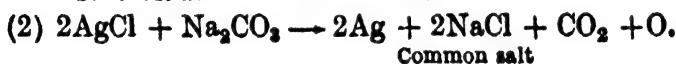
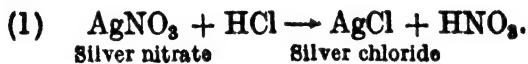
*Exp. 166.* Put 5-10 grams of silver scrap into a beaker, add 6 c.c. of distilled water and 4 c.c. of strong nitric acid for every gram of the alloy to be dissolved. Cover the beaker with a clock-glass, and heat it carefully on the hot plate. If, when the metal is dissolved, the solution is not quite clear, filter it into another beaker, and well wash the filter with distilled water, collecting the washings in the second beaker. Add water to the filtrate, if necessary, until the beaker is half full, and raise it to boiling on gauze over the Bunsen. Lower the flame to keep the solution gently boiling, and add slowly 1 c.c. of strong hydrochloric acid per gram of the alloy taken. Continue the boiling for two or three minutes, remove from the gauze, and when the white, curdy precipitate has settled add a drop or two of the strong acid to see that all the silver is precipitated. If not, a little more acid must be added and the solution well stirred with a glass rod. If the solution remains clear, filter, and transfer the whole of the precipitate to the filter. Thoroughly wash the precipitate on the filter with hot water, and put the funnel, together with the filter, in a drying cone on the hot plate.

Heat a clay crucible to a red heat in the furnace, shoot 5 grams of borax into it, and, when the salt has melted, grip the crucible in the tongs, withdraw it from the fire, and cause the molten borax to run all over the surface. This glazing prevents the absorption of silver chloride by the walls of the crucible. Place the dry silver chloride and filter paper on the weight pan and counterpoise it with sodium carbonate. Transfer the carbonate

to the glazed crucible, and tip the chloride from the filter paper into it ; remove as much of the precipitate as possible from the paper to the crucible, then screw up the paper, and put it on top of the mixture. Place the crucible back in the fire, and when the mixture has settled down into tranquil fusion, pour the contents of the crucible into a round mould. Break away the slag, which is a mixture of common salt and unchanged carbonate, clean the silver button, and weigh it. The button should be rolled into a strip, well scrubbed with pumice powder, swilled, and dried. It is then ready for use.

The crucible should be watched while the fusion is taking place, as gas is liberated from the carbonate during the reduction of the chloride. This may cause the mixture to boil over, if the crucible is more than half full. A little common salt sprinkled on the surface will modify this action, and prevent loss.

The changes taking place in the precipitation and reduction of the silver chloride are shown in the equations :



The determination of the percentage of silver in an alloy may be made by modifying the first part of the above.

*Exp. 167.* Weigh exactly about .5 gram of the alloy, and dissolve it in 6 c.c. of water and 4 c.c. of strong nitric acid. Precipitate the silver as chloride. Fold two filter papers and put them in a filter funnel in the air oven to dry. When dry, place one in each pan of the balance, and counterpoise them, cutting the apex off the heavier one and then snipping the one or the other at the top until they counterpoise. Then put the complete filter into the one with the apex removed, and open out into a complete filter with four folds on each side. Place the double filter in the funnel, and filter the chloride. After thoroughly

washing the precipitate place the funnel in the air oven heated to about 100°C. When dry, separate the filters, put the outside one on the weight pan and the other on the weighing pan, and counterpoise. The added weights are equal to the weight of the silver chloride. Do not take the result on the first weighing, but repeat the drying until the weight is constant.

The weight of silver in the chloride is found from the relation  $\frac{\text{Ag}}{\text{AgCl}} = \frac{108}{108 + 35.5} = .753$ . If then the weight of chloride obtained is multiplied by .753, the weight of silver in it is determined, and this multiplied by 200 gives the percentage of silver in the alloy, since .5 gram was taken.

#### EXAMPLE

$$\begin{aligned}\text{Weight of alloy taken} &= 0.500 \\ \text{Weight of chloride obtained} &= 0.603 \\ \therefore \text{Silver per cent. in alloy} &= 90.62\end{aligned}$$

**Volumetric Assay of Silver Alloys.** The term gravimetric is applied to the method described above, but the assay may be made much more rapidly by the use of standard solutions measured from a burette. There are several volumetric methods for silver assay, but only the one mostly used in assay offices will be described here.

**The Thiocyanate Method.** Potassium thiocyanate, KCNS, is a crystalline salt which dissolves readily in water to a colourless solution. If a single drop of this solution is added to a solution of ferric sulphate containing nitric acid, the solution is discoloured, but if silver is also present in the iron solution no permanent discolouration takes place until the whole of the silver has been converted into insoluble thiocyanate. When this is effected, a single drop produces discolouration. This is the principle of the process.

**Standard Silver Solution.** Weigh exactly 5 grams of pure silver, dissolve in 30 c.c. of distilled water and 20 c.c. of strong nitric acid ; boil to expel red fumes ; allow to cool ; pour into a 500 c.c. measuring flask ; well wash the beaker with distilled water and add the washings to the flask ; add distilled water up the mark, put in the stopper, and thoroughly mix the contents of the flask. The solution should then be transferred to a clean stoppered bottle and marked

$$1 \text{ c.c.} = .01 \text{ gram of silver.}$$

**Ferric Sulphate Solution.** Dissolve about 5 grams of ferrous sulphate,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , in 100 c.c. of distilled water ; add strong nitric acid, a little at a time, to the hot solution until it changes colour, and boil to expel red fumes. When cold, transfer to a stoppered bottle and label it ferric sulphate solution.

**Pure Nitric Acid Solution.** Add 150 c.c. of strong nitric acid to 50 c.c. of distilled water in a beaker. Boil for a minute or two, and when cold pour into a stoppered bottle, label it nitric acid solution, and keep in a dark corner of the cupboard.

**Standard Thiocyanate Solution.** Dissolve 5 grams of potassium thiocyanate in distilled water in a 500 c.c. flask and make up to the mark. Thoroughly mix and transfer the solution to a stoppered bottle. Fill up the burette A, Fig. 12, to the mark after washing it out with distilled water and rinsing it with a small quantity of the thiocyanate solution. Transfer 25 c.c. of the standard silver solution to a conical flask by means of a 25 c.c. pipette. Add about 25 c.c. of distilled water, 5 c.c. of the ferric sulphate solution, and 10 c.c. of the nitric acid solution. Run in the thio-

cyanate from the burette through the tap B, shaking the flask and allowing the white silver thiocyanate to settle between each addition, gradually reducing the quantity added until one drop produces a brownish colour in the solution which does not disappear on shaking. The thiocyanate is now very slightly in excess, and the titration is finished. Read the burette and note the volume of solution added.

The volume of the standard solution added is equivalent to the weight of silver, that is .25 gram, in the 25 c.c. taken. If then the weight of silver is divided by the number of c.c. of thiocyanate used, the value of 1 c.c. in terms of silver is obtained. Volume of solution = 24.3 c.c.

$$\text{Value of 1 c.c.} = \frac{\text{weight of silver}}{\text{volume of solution}} = \frac{.25}{24.3} = .0103 \text{ gram of silver.}$$

This value should be marked on the bottle.

*Exp. 168.* Weigh exactly .25 gram of standard silver; transfer it to a conical flask, and add 8 c.c. of water and 5 c.c. of strong nitric acid. Place the flask on the hot plate, and when the metal has dissolved, add a little water and boil gently until the red fumes are dispersed. Add 20 c.c. of water, 5 c.c. of the ferric sulphate solution, and 10 c.c. of the nitric acid solution. Fill the burette up to the mark with the thiocyanate solution, level it, and run it into the silver solution with the precautions described above. Read the burette when the brownish coloration is permanent.

#### EXAMPLE

$$\begin{array}{rcl} \text{Weight of alloy taken} & = & .25 \text{ gram} \\ \text{Volume of thiocyanate solution required} & = & 22.45 \text{ c.c.} \end{array}$$

$$\text{Then } \frac{22.45 \times .0103}{.25} \times 100 = 92.48 \text{ per cent. silver.}$$

Two assays should be made, and the mean of the results taken.

The method is largely adopted in assay offices for hall-marking, as it is very rapid when the various solutions are prepared. It is not necessary to make any calculation, for if an exact quantity of the alloy is weighed and a volume of the thiocyanate just insufficient to precipitate the whole of the silver is added, no coloration takes place if it is up to standard. But if the supernatent liquid is coloured when the precipitate has settled, the alloy is rejected for hall-marking. In this case a second assay is made to confirm the first result.

**Gold and its Alloys.** As already mentioned, pure gold is too soft for ordinary purposes and is only used for colour effects on ornamental work. Ordinary alloys contain copper and silver in varying quantities, and as they are for the most part solid solutions their working properties are excellent. The quality of a gold alloy is expressed in carats, 24 carats being 1000 fine. Thus a 15-carat alloy contains  $\frac{15}{24}$  gold. On account of the cost of the principal metal it is not possible to make gold alloys in the laboratory, but exercises in alloying the common metals may be selected from Chapter VII.

*Exp. 169.* Weigh accurately about .2 gram of fine gold, and .2 gram of copper; wrap the two metals in 3.5 grams of lead free from silver, and cupel the packet on a smooth cupel. Remove any adhering matter from the underside of the gold bead by brushing with a hard toothbrush, and weigh it. It should weigh very nearly the same as before cupellation, for the whole of the copper is removed and there is very little fire loss.

*Exp. 170.* Weigh .5 gram of fine silver; wrap it, together with the gold bead, in 2 grams of lead, and cupel the packet. The gold-silver alloy should weigh about .7 gram.

**The Parting of Gold and Silver.** All the common metals usually associated with gold in its alloys are

removed by cupellation. Silver, however, simply alloys with it in the process, and another operation is necessary for their separation. The only method is that known as parting, which depends upon the fact that if sufficient silver is present in the alloy it can be completely dissolved out by nitric acid or by strong sulphuric acid. For assay work nitric acid only is used. There must be at least two and a half times as much silver as gold in the alloy for sufficiently perfect parting. Unless it is known for certain that silver is absent this process must be carried through with every gold alloy.

*Exp. 171.* Take the bead obtained in the last experiment and pass it through the rolls until it is flattened into a strip about 1 in. long. Heat the strip to low redness in the Bunsen flame. To do this, hold one end in the pliers and heat the other in the flame, then reverse the process. This softens the metal, and also burns off any grease adhering to it from the rolls. Make the strip into a loose coil, and drop it into a test-tube about a quarter full of dilute nitric acid (1-1). Place the tube in water boiling in a beaker on gauze. When the action has ceased, and the red fumes have disappeared, pour off the solution, which contains nearly all the silver, into the residue bottle, or into another beaker. Add enough strong nitric acid to the test-tube to well cover the cornet, and return the tube to the water bath; allow to remain for 5 minutes. Pour off as before, and well wash the cornet with distilled water, pouring off after each addition. Then fill the tube with water, and invert it in a porcelain crucible also filled with water. The gold falls readily into the crucible, and when it has all collected, remove the tube. Pour off the water from the crucible, and soak up what is left with a piece of filter paper; dry the crucible, and heat it over the Bunsen flame until the gold "colours." Transfer the gold to a counterpoised watch-glass and weigh it. The weight of the gold should be very nearly the same as that of the fine gold taken.

In the assay for hall-marking it is necessary to make a correction for fire loss, which would in many cases

cause the assay to come out too low. With a number of assays there are usually two checks made up with the same weight of pure gold as the alloys under test are supposed to contain. These are placed in position in the muffle at the discretion of the assayer. What happens to them is supposed to happen to the other assays, and any loss they sustain is added to the others in determining whether they are up to standard. The loss may be due to volatilization of a small quantity of gold or to absorption into the cupel. Sometimes the gold, after parting, may weigh a little heavier, owing to imperfect parting of the silver. In this case the increase is subtracted.

The articles to be hall-marked are scraped in various parts, and one or more assay quantities of 5 grains each taken. These are weighed, and two and a half times the weight of fine silver to gold present is added. The weight of lead for cupellation is determined by the carat of the alloy to be assayed. Thus the weight for 12-carat is  $2\frac{1}{2} \times 16 = 40$  grains. Part of this is used for making the assay packet, and part is placed on the cupel before the addition of the packet. The gold-silver bead obtained is made into a cornet, placed in a platinum thimble, and immersed in a bath of hot nitric acid. It is then passed on to a similar bath, and finally thoroughly washed, dried, heated, and weighed. A number of similar assays are worked off at the same time.

*Exp. 172.* Determine the gold in one or more carat alloys. Follow the instructions given in the last experiment. Check-assays need not be made.

**Assay of Gold Ores.** The precious metal is present in these ores in the free state, and may be obtained on

the small scale by concentration in lead, which is then cupelled to separate the gold.

*Exp. 173.* Take 40 grams of the finely powdered and sifted ore, 45 grams of red lead, 1.5 grams of charcoal, 30 grams of sodium carbonate, and 15 grams of borax. Well mix the red lead with the charcoal, add the ore and fluxes, and mix again. Transfer the mixture to a clay crucible, which should not be more than two-thirds full, and sprinkle a little borax over it. Place the crucible in a moderate fire, and allow the mixture to come slowly to a red heat; then put the crucible lower in the fire so that it is completely surrounded with hot coke. When quite molten and tranquil, pour the melt into a round mould. Break the slag away from the lead button, and well hammer it on the edge to test if it is soft. If it is hard, it should be scorified before cupellation.

Transfer the lead to a large cupel in the muffle. When finished, take off the bead and weigh it. Add to it  $2\frac{1}{2}$  times its weight of silver and cupel again with 2 grams of lead. Make the bead into a cornet, and part it as already described. Weigh the fine gold, and calculate the percentages of gold and silver in the ore.

#### EXAMPLE

Weight of gold-silver bead	=	.011 gram
"    gold	=	.008
"    silver	=	.003

$$\text{Percentage of gold} = .008 \times \frac{1}{2} = .02$$

$$\text{    "    silver} = .003 \times \frac{1}{2} = .0075.$$

A second assay of the same ore should be made as a check upon the first.

**Lemel.** Filings and other finely divided material rich in gold produced by the goldsmith and jeweller are known as lemel, and are carefully collected. The principal impurity consists of particles of iron from the files and other tools used in the manipulation of the metal, and these must be completely removed by a magnet. The lemel is then mixed with one-sixth

its weight of sodium carbonate and one-fourth its weight of common salt. The mixture is put into a skittle pot, which should not be more than three-fourths full and placed well down in a good fire. It should be carefully watched, and if it shows signs of boiling over a little more salt should be added. When the mass has settled down into tranquil fusion the pot is withdrawn, allowed to get cold, broken, and the button of metal separated from the slag. The button is then melted with borax as flux, together with a little potassium nitrate added when the metal has run down. A partial refining is thus effected and a good ingot obtained. The ingot is then assayed, and either made up again or sold to the bullion dealer.

As lead is not common material in the laboratory the method may be worked through with filings or turnings of copper alloys.

*Exp. 174.* Take about 50 grams of brass or gun-metal turnings and remove iron with the magnet. Weigh 50 grams of cleaned metal; mix with it 8 grams of sodium carbonate and 5 grams of common salt, and melt the mixture in a clay crucible. When the melt is finished, withdraw the crucible; let it get cold, and break it to obtain the button of alloy. Weigh the button, and remelt it under a little charcoal. Pour the molten alloy into the strip mould.

**Jewellers' Sweep.** The material indicated by this name consists chiefly of the floor sweepings of the workshop and other places where particles of metal are likely to accumulate. It contains sufficient gold and silver to make it valuable. The sweepings are burnt in an iron pan to remove bits of paper, fragments of wood, and other combustible matter. The residue is pounded in a mortar and sifted before being put into the sweep bin. Other additions are parts of old

crucibles, stirrers, and other débris likely to contain the precious metals. These are also pounded and sifted before being added to the bin. When sufficient has been collected it is thoroughly well mixed and sampled for assay. The sweep that finds its way into the laboratory is of this character.

*Exp. 175.* Take of sweep, 20 grams; sodium carbonate, 20; borax, 20; common salt, 15; potassium nitrate, 3; red lead, 35; charcoal, 2. Well mix the red lead and charcoal, then mix in the sweep, and finally the fluxes. Transfer the mixture to a clay crucible which should not be more than two-thirds full. If preferred, one-third of the common salt may be reserved and sprinkled over the top of the mixture. Run down in a good fire, and when the mass is tranquil pour into a round mould. When sufficiently cold break away the slag, and weigh the lead button. Hammer the button on the edge, and if it is soft cupel it straight away; but if hard from the presence of metallic impurities, it saves time to scoria before cupellation. Weigh the gold-silver button and cupel it with  $2\frac{1}{2}$  times its weight of silver and 2 grams of lead. Part the gold and silver (Exp. 171) and weigh the gold.

It is usual to calculate the gold and silver in grains per cwt. To do this, multiply the weight of sweep taken and the weight of gold and silver obtained in grams by 15.432 to reduce to grains. Then since there are 784,000 grains to the cwt. the calculation is easy.

The lead oxide which is absorbed by the cupel carries with it some gold and silver and this, in part, accounts for the slight loss of metal which occurs during cupellation. The more lead there is to remove the greater the loss. Too high a temperature acts in the same direction. The presence of the precious metals in the old cupels may be easily demonstrated by obtaining the lead from them and cupelling it.

*Exp. 176.* Collect two or three well-saturated cupels which have been used for gold assays; break away the parts which show

no signs of lead oxide, and crush the remainder to a fine powder. Well mix, and weigh 30 grams of the powder, 40 grams of fluor-spar, 40 grams of borax, and 40 grams of sodium carbonate. Mix the cupel powder with 1.5 grams of charcoal, and then mix in the fluxes, reserving half the borax to put on the top. Transfer the mixture to the crucible used for the sweep assay, and melt in a good fire, but do not let the temperature get up too rapidly. When tranquil, pour into a button mould. Separate the lead from the slag, and ascertain if it is soft by hammering. Cupel the bead and examine the cupel for the silver bead.

The bead is usually not large enough to weigh, but if it is put into a test-tube and heated with a little dilute nitric acid, a black speck of undissolved gold will be noticed in the bottom of the tube.

**Gold Solutions.** The common solvent for gold is aqua regia, a mixture of hydrochloric and nitric acids in the proportion of 3-1 by volume. Gold chloride,  $\text{AuCl}_3$ , is formed by the action of free chlorine liberated in the acid mixture. The chloride may be obtained in the pure state by evaporating the solution on the water-bath, with several additions of water to remove the excess of acids. Metallic gold may be obtained from the chloride solution by the addition of ferrous sulphate. The metal is thus thrown down as a dark brown precipitate which is collected, well washed, and melted. This is the method adopted for precipitating the gold from "colour" water obtained as a residue in the "colouring" process which is largely used for finishing articles made from the higher qualities of carat gold. In this process potassium nitrate takes the place of nitric acid in the "colour" mixture, but the change on the surface of articles is still brought about by free chlorine, which dissolves out a little gold in addition to the alloying metals. Hence the necessity of treating the residues for the gold they contain.

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